

ORGANIC CHEMISTRY OF BIVALENT SULFUR

VOLUME II

by

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CHAPTER 1. ---

Occurrences and Preparation of Sulfides

Introduction

The alkyl sulfides, RSR' , are the analogs of the ethers, ROR' . There are the same formal relationships between these two classes of compounds as between mercaptans and alcohols. In other respects, the differences are more notable than the similarities.

Alcohols are found widely distributed in natural products, either as such or as esters from which they are easily liberated. Not so with ethers, other than methyl. Sulfides are widely distributed in nature (though in small quantities), while mercaptans are comparatively rare. The synthesis of sulfides is far easier than that of ethers and, consequently, far more of them are known.*

* Nearly everyone who ever made a sulfide gave some information about its properties and said something about its reactions. The authors cited in this and in the following two chapters are largely the same. To save space, the literature cited in the three chapters has been combined and placed at the end of Chapter 3.

Occurrence

Methyl sulfide is found in African and Reunion geranium oil and in peppermint oil.^{20, 363} The same sulfide is evolved from *Polysiphonia fastigiata*.^{278, 525} It is in the odor of eucalyptus.¹⁰⁸¹ Allyl sulfide is responsible for the odor of garlic.^{73, 528, 1006, 1310, 1357, 1388} Vinyl sulfide and polymeric vinyl sulfides are also present.¹⁴¹⁵ Five minutes after eating 2 g. of garlic, the expired air contains 0.0028 to 0.0035 mg. per liter.⁵²⁸ Ethyl sulfide is also present.⁷³ Vinyl sulfide has been isolated from *allium ursinum*.¹¹⁷¹ The antibacterial principle of *allium sativum* appears to be $C_3H_5SO \cdot SC_3H_5$.^{267, 268, 1233} Crotyl sulfide has been isolated from the skunk secretion.¹²³¹ An unstable sulfur compound, possibly a sulfide, has been obtained from asafoetida oil.⁵⁸⁵ γ -Methylmercaptopropanol, $CH_3SCH_2CH_2CH_2OH$, has been distilled out of soy sauce.⁷ The methyl ester of a sulfide acid, $MeSCH_2CH_2CO_2Me$, b₁₁ 69°, has been isolated from pineapples in which it is present to the extent of about 1 part in 1,000,000.⁵²² With these may be put 4-methylsulfoxide-butenyl-3-cyanide, $MeSOCH:CHCH_2CH_2CN$, which has been isolated from the seeds of *Raphanus sativus*, var. *alba*.¹¹⁴⁵

Alkyl sulfides, which are not primary products, are found. Thus methyl sulfide appears to be in the oil which is a by-product in making sulfate pulp from spruce though it is not present in the original wood.⁵³⁴ There was found 0.54 kg. of it per ton of cellulose in the waste gases from the sulfite process.⁴⁹⁶ It is present in the blow gases from Kraft pulp.⁴¹⁴ Alkyl sulfides can be isolated from "black liquor."^{103, 1104} Methyl sulfide from this source has been sold for odorizing natural gas. Means for increasing the yield have been suggested.^{527, 5} In a recent commercial process, the black liquor is concentrated to 50% solids and mixed with sulfur compounds, such as Kraft recovery smelt, and heated to 380°F. and later to 450°F. About half of the methoxyls of the lignin are said to be converted to methyl sulfide.^{560, 5} A sulfide, which can be isolated from dog urine, comes from the decomposition of methyldiethylsulfonium hydroxide, or one of its salts. This, in turn, is a product of metabolism.^{1, 287, 378, 1132, 1380} Ethyl sulfide is supposed to be produced from cystine by *B. coli*.⁷²¹

From the alcohol extract of yeast a thio-sugar has been isolated. This appears to be a ketohexose in which the hydroxyl on the

first carbon has been replaced by $-SMe$.^{1248, 1249} The saponification of fatty matter from the adrenal cortex gave, along with glycerol, a small amount of a trihydroxysulfide or a dihydroxysulfide.¹⁰⁷⁸ Alcohol from beet molasses and from fruit juices often contains mercaptans and sulfides, but these may be derived from sulfur dioxide used in treatment of the raw products.⁸⁴⁹

Certain molds of the penicillin group convert alkyl disulfides, $RS\cdot SR$, into methyl alkyl sulfides, $MeSR$, and sodium selenite and tellurite, Na_2SeO_3 and Na_2TeO_3 , into methyl selenide and telluride, Me_2Se and Me_2Te .^{120a, 122, 270b, 271, 274}

Methyl, ethyl, methyl-ethyl, ethyl-propyl, propyl, butyl, butyl-amyl, amyl, and hexyl sulfides have been reported in petroleum distillates.^{816a, 816c, 817a, 817b, 818a, 818b, 933, 1280} Some of these may have resulted from decomposition of sulfur compounds originally present or from the addition of mercaptans to unsaturates, such as ethylene, propylene, and butylene. This remark applies with greater force to the alkyl sulfides found in cracked distillates.^{157, 190b, 381, 665, 831, 928, 1144} The crude oil of the Cushing field contains alkyl sulfides.⁴⁴² Phenyl sulfide has been reported in mineral oil.^{270a} Of the sulfur in Nebitdag crude oil 37.3% is in sulfides, 4.4% in mercaptans, and 26.3% in disulfides.¹³⁹⁰

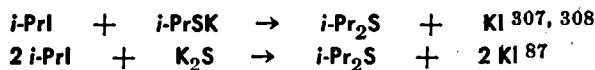
Sulfides have been isolated from shale oil,^{71, 1275} low-temperature tar,^{467, 1157} and from illuminating gas.¹³⁷⁷ Divinyl sulfide is found in crude acetylene.⁶⁷⁹ The presence of organic sulfides in coal is indicated by the reaction with methyl iodide.¹⁰²⁰ Methyl sulfide has been found in coal tar.^{833a}

History

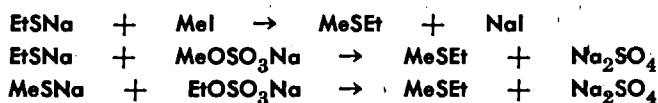
The history of alkyl sulfides is practically identical with that of mercaptans. After getting a reaction product from potassium hydrosulfide, it was natural to try the neutral potassium sulfide.¹³⁹⁷ Besides, as was brought out in the chapter on mercaptans, it is difficult to make a mercaptan without obtaining some of the sulfide as a by-product.

Döbereiner heated alcohol, hydrochloric acid, and ferrous sulfide in a retort and got an evil-smelling liquid.³⁶⁹ Zeise heated barium ethyl sulfate and isolated ethyl sulfide.¹³⁹⁷ Regnault made methyl and ethyl sulfides from methyl and ethyl chlorides and alcoholic potassium sulfide.¹⁰⁷⁷ Balard prepared *i*-amyl sulfide similarly.⁶⁴ Ethyl sulfide was obtained from potassium

ethyl sulfate and sodium thiosulfate.⁴⁵³ Cetyl sulfide was prepared from the chloride by Fridau⁴⁴⁶ and propyl sulfide from the iodide by Cahours^{245b} and Winssinger.^{1376a} The synthesis of *i*-propyl sulfide in two ways showed clearly its relation to the mercaptan:

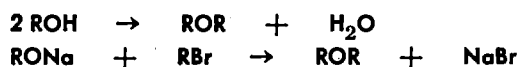


The mixed methyl-ethyl and *i*-amyl-ethyl sulfides were made by Carius.²⁵³ Other mixed sulfides soon followed.^{87, 740, 950, 1137a} The methyl-ethyl sulfide was made in three ways: 700a, 700d



The Preparation of Sulfides

For ethers we have two general methods: the catalytic dehydration of an alcohol and the Williamson synthesis from an alkyl halide and a metal alcoholate:



The dehydration may be effected by passing the alcohol vapor over a solid catalyst at an elevated temperature. Ethers are commonly manufactured by boiling the alcohols with dilute sulfuric acid. The reaction involves the breaking of the hydrogen-oxygen bond in one molecule of the alcohol and the carbon-oxygen bond in the other. Therefore, the yield of the mixed ether is high when one of the alcohols is tertiary.⁹⁴⁶

The preparation of a sulfide from a mercaptan by acid catalysis is not favored since the carbon-sulfur bond is not easily broken:

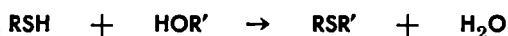


This reaction does take place when a mercaptan is passed over a catalyst,^{454, 780, 1128, 1163, 1164, 1165, 1297} such as cadmium or zinc sulfide, at 300° or above.^{454, 1163, 1164, 1367} *t*-Butyl mercaptan is converted to *t*-butyl sulfide by a Friedel-Crafts catalyst, aluminum chloride, hydrofluoric acid, or boron trifluoride.⁸⁷⁷

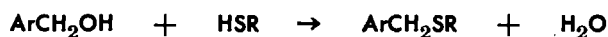
Alkyl sulfides are formed along with mercaptans when alco-

hols and hydrogen sulfide are passed over a catalyst, such as thoria.^{731, 1128} The same is true when an alcohol vapor is passed over heated aluminum sulfide.^{768, 769}

A sulfide can result from an acid-catalyzed reaction of a mercaptan and an alcohol, especially when the hydroxyl of the alcohol is readily eliminated:^{69, 237, 601}



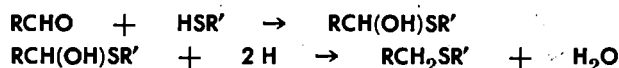
t-Butyl mercaptan is added slowly to a well-cooled solution of *t*-butyl alcohol in 78% sulfuric acid. After standing for some time, this solution is poured onto ice to give 87% of *t*-butyl sulfide.^{412a} A substituted benzyl alcohol condenses with a mercaptan in the presence of an acid:^{121, 1121}



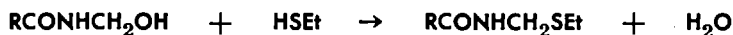
Triphenylcarbinol gives particularly good yields.^{429, 516} Thiophenol and ethanol vapors passed over a thoria-alumina catalyst at 350° give 62% of PhSEt.⁵³⁹

In the presence of alkali, thiophenol reacts with an alkyl phenyl ether.^{619a, 767}

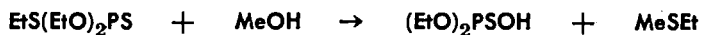
When an aldehyde or ketone is hydrogenated in the presence of a mercaptan, the product is a sulfide. This may be imagined as the reduction of the hemimercaptal:^{1190d}



A methylolamide reacts with a mercaptan:³⁹



Ethyl dithiophosphate and methanol give methyl ethyl sulfide:²⁵³



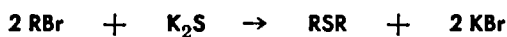
Ethyl sulfide is formed by the reaction of zinc ethyl on thionyl chloride:^{477b}



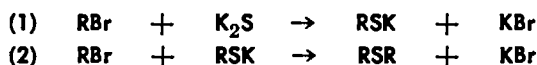
Grignard reagents react similarly.⁹⁵¹ Betaine and butyl disulfide give methyl butyl sulfide.²⁷⁹ Menthone in hydrochloric acid solution is turned into menthenyl sulfide by hydrogen sulfide.^{455c}

SYMMETRICAL ALKYL SULFIDES

As was mentioned before, the preparation of these sulfides has been covered to some extent in Volume I on mercaptans. As was brought out there, the most commonly employed methods of making mercaptans yield more or less of the alkyl sulfides along with the mercaptans. The proportions of the two in the product depend on the degree of saturation of the alkali by hydrogen sulfide and on other conditions. The formation of a sulfide is usually written:



It is more accurate to write it as two consecutive reactions:



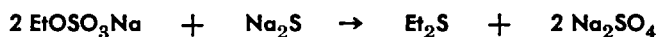
At any time during the operation, potassium sulfide and mercaptide are present and the alkyl bromide may react with either. The potassium mercaptide is readily hydrolyzed and some of the mercaptan may escape, in which case the second reaction cannot be completed. To get the maximum yield of the alkyl sulfide, only a slight excess of the alkali sulfide should be used and the mercaptan should be kept in the solution.

For preparing symmetrical alkyl sulfides, alkyl chlorides, bromides, or iodides may be used, the choice being determined by considerations of cost and convenience. Methyl and ethyl iodides have been employed in the laboratory since the corresponding chlorides and bromides are inconveniently volatile. These and other iodides were favorites with the early chemists.^{82, 245b, 247, 506, 596, 804, 978a, 1091} From propyl on up the alkyl bromides, which are readily prepared and react satisfactorily, have been used in laboratory preparations.^{87, 110, 161b, 685, 760b, 795, 1297, 1376b} The reaction of an alkyl bromide with sodium thiophenate in alcohol solution is so rapid that ethyl bromide may be used in spite of its low boiling point, 38°. ^{961b} An early chemist went to the trouble of using ethyl iodide in a sealed tube at 120°. ⁸⁷ Alkyl chlorides have been employed since early times and have come to be standard for large-scale manufacture where price per pound is a determining factor. The more volatile and less reactive chlorides can be treated in autoclaves.^{105c, 348a, 740, 902, 915b, 960c, 984, 1133, 1328b} Benzyl

chloride and its homologs, such as PhCHClEt , are favored on account of their reactivity and availability.^{416, 606, 832a, 976a, 1154}

Unsaturated halides, such as hexenyl,³⁶⁰ crotonyl,²⁸² butadienyl,²⁵⁸ and isobutenyl⁵¹⁸ chlorides, react satisfactorily with metal sulfides.

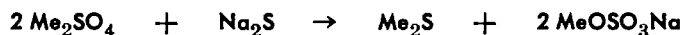
The monoalkyl alkali sulfates, such as $\text{MeO}\cdot\text{SO}_3\text{K}$ and $\text{EtO}\cdot\text{SO}_3\text{Na}$, which are normally tardy alkylating agents, can be employed with alkali compounds of bivalent sulfur:



The superreactivity of the alkali sulfur compounds makes up for the low reactivity of the alkyl sulfates. Sulfates of higher alcohols may be used.^{512, 570a, 586, 700b, 700d, 790}

The alkyl metal sulfates have been used in making alkyl selenides^{653, 662, 1002} and tellurides.^{662, 836, 1378, 1379}

The dialkyl sulfates, when available, are preferred to the alkyl alkali sulfates on account of their superior reactivity:^{140, 694}



In this, only half of the methyl groups is utilized. The other half may be made to react with additional sodium sulfide by raising the temperature or prolonging the time of heating. The alkyl esters of *p*-toluene sulfonic acid are active alkylating agents and can be used for making alkyl sulfides.^{377b, 1396}

In the academic laboratory, alcohol is the commonly used solvent. It is convenient to mix the alkyl halide with the alcohol and add the hydrated sodium sulfide, $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$, dissolved in a minimum amount of water, in portions or dropwise, with shaking or stirring. Even at room temperature, the reaction is rapid, as shown by the precipitation of sodium halide. It can be regulated by the rate of addition of the sulfide solution. It is customary to heat the reaction mixture at the end to insure the completion of the reaction. The alcoholic solution is filtered to get rid of the precipitated alkali salt and the alcohol is distilled off. The crude alkyl sulfide may be filtered to get rid of any sediment. Any of the sulfide adhering to the filter paper or to containers may be collected in a little ether and added to the main portion.

In commercial operations, water must serve as the solvent. The solubility of the alkyl halides in water is small but, on account of the high reactivity of alkali-sulfur compounds, it is

sufficient, at least for the lower halides. In many cases it is convenient to add the alkyl halide dropwise to the stirred solution of the sodium sulfide kept at 70 to 80°. The heating and the rate of addition of the alkyl halide are so adjusted that the temperature remains nearly constant. The addition of about 0.04 mole of magnesium chloride and an equivalent amount of sodium hydroxide, to precipitate magnesium hydroxide, and a dispersing agent may facilitate the reaction.⁹⁷⁹

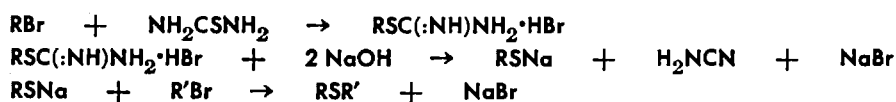
If the alkyl sulfide boils below 200°, it is best to steam-distill it out. The sulfide is separated from the water layer of the distillate. The small amount of the sulfide in the water layer is steam-distilled out, separated, and added to the main portion. If the sulfide is not sufficiently volatile to be recovered in this way, it is taken off in a separatory funnel.

As explained before, the crude alkyl sulfide may contain more or less of the mercaptan. It seemed obvious to early chemists to take this out by extraction with aqueous alkali. As explained in the chapter on mercaptans, this treatment is inefficient for the lower mercaptans and entirely useless for the higher. Furthermore, it is harmful since mercaptans are oxidised to disulfides by air in the presence of alkali. It is far better to rely on fractionation. In crude butyl sulfide, the probable impurities are butyl mercaptan, b. 98°, and butyl bromide, b. 101.6°. As butyl sulfide boils at 182°, its separation from these is easy, even with a simple column. Any water which may be present goes over with the first few drops of distillate. Distillation is by far the best way to dry liquids which do not mix with water. The small amount of cloudy distillate is caught by itself and separated.

Heating an alkyl sulfide with copper powder in a sealed tube⁴²⁷ or distilling it over copper powder^{820, 1382} has been recommended for the elimination of traces of mercaptans.

Alkyl sulfides which are solids can be prepared on a small scale. Thus, 1 g. of myristyl bromide is added to a solution of 0.45 g. crystalline sodium sulfide (calculated 0.43 g.) in 5 cc. alcohol in a test tube. This is kept at about 70° for several hours and 3 cc. of hot water added. The tube is corked and shaken vigorously in a stream of cold water. The myristyl sulfide is filtered off and dried; yield 0.7 g. or 90%, m. 53.8°. ¹⁰⁸⁰

A convenient way to make an alkyl sulfide is to react an alkyl halide with a mercaptan as it is being set free from an isothiuronium salt:

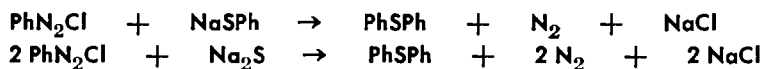


The cyanamide, which dimerizes, does not interfere with the reaction of the second alkyl halide with the sodium mercaptide. This is the procedure: Reflux the alkyl halide with a slight excess of thiourea until the reaction is complete. Have ready 2.2 equivalents of sodium hydroxide dissolved in twice its weight of water. Remove the source of heat from the reaction flask and run the alkali solution in from a dropping funnel at such a rate that the reaction does not become violent. As this goes in, the isothiuronium base usually starts to separate as a white mass which soon disappears. By the time half of the alkali is in, the mercaptan separates as a layer. Heating may be required to complete this reaction. Add the balance of the alkali and run in an equivalent of alkyl halide from the same dropping funnel at such a rate that the reaction is not violent. Heat for a few minutes and steam-distill out the sulfide if it boils below 200°; otherwise separate it as a layer or cool and crystallize it out.¹⁰⁷⁹ Or the halide may be added to the isothiuronium salt solution and then the alkali put in.⁶³

This works equally well for a symmetrical and an unsymmetrical alkyl sulfide. In the first case, the alkyl halide used to react with the mercaptan is the same as that used in the first part while in the other it is different. If the second halide is easily hydrolyzed, it is better to cool the mixture, after liberating the mercaptan, in an ice bath if necessary, before adding the second halide.

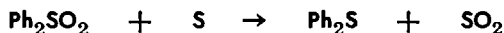
SYMMETRICAL AROMATIC SULFIDES

Since aromatic halides, except in special cases, are unreactive, aromatic sulfides cannot be prepared by the method that serves so well for aliphatic sulfides. They can be made from diazonium salts: 145, 508, 1044, 1400



An aromatic sulfoxide may be reduced to a sulfide by sodium and alcohol.⁴⁶⁶ Diphenyl sulfoxide has been reduced.¹¹⁹⁸ This sulfoxide, refluxed with sodamide in benzene, yields biphenylene sulfide.^{329, 1152}

An aromatic sulfone can be reduced to the sulfide by zinc and acid or by lithium aluminum hydride.¹⁵⁸ Diphenyl sulfone can be reduced by sulfur: ^{483.5}

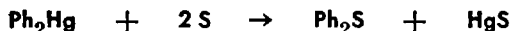


It is a curious fact that when this sulfone is heated with selenium, the product is diphenyl selenide.^{478, 726a, 729, 792} An unsymmetrical sulfone gives an unsymmetrical selenide.⁴⁷⁸ However, when diphenyl selenide is heated to 300° with one equivalent of sulfur, 95% of the selenium is replaced by sulfur. The same happens with diphenyl telluride.⁷²⁸

Some diphenyl sulfide is formed in the pyrolysis of sodium benzenesulfonate.¹²²⁸ The distillation of potassium naphthalene-sulfonate with potassium thiocyanate gives naphthyl sulfide.²⁵ Diphenyl and dinaphthyl sulfides are obtained by the pyrolysis of the corresponding mercaptides: ^{723a, 723b, 727}



Mercury aryls are converted to sulfides, selenides, or tellurides by heating with the proper elements: ^{726b, 1398}



The halogen in *o*-nitrochlorobenzene and in *p*-nitrochlorobenzene is activated by the nitro group so that these halides react regularly with sodium sulfide to give the corresponding nitrophenyl sulfides.^{873, 865, 875} Under certain conditions, the nitro group may be reduced by the sodium sulfide so that the product is an aminophenyl sulfide.^{464, 591} In some cases, one of several nitro groups may be replaced by the sulfur.^{590b, 680, 789, 1206} The sulfide may be obtained by the reaction of *p*-nitrochlorobenzene with sodium thiosulfate.^{8, 339} 2,4-Dinitrochlorobenzene approaches an alkyl halide in reactivity.⁴⁹⁷ It gives tetranitrodiphenyl sulfide with sodium sulfide.⁵⁸⁷ It reacts similarly with sodium selenide.^{88b} 1,4-Nitrochloronaphthalene reacts similarly.⁵⁸⁹ In alcohol solution, the reaction is complete in 8 hours at 20°. With the other isomers, it is much slower.⁸⁰⁸ 1,6,8,2-(NO₂)₃C₁₀H₄Cl reacts well.⁶⁶³ 5-Nitro-2-chlorofurane¹¹⁷⁸ and 5-nitro-2-bromofurane⁵²⁶ react satisfactorily with sodium sulfide to give 5,5'-dinitrofuryl sulfide.

In the absence of an activating group, such as the nitro, phenyl chloride is inert, but it can be made to react with sodium sulfide

by heating to 350 to 360° under 180 to 190 atm.^{1331, 1332a} Phenyl bromide reacts at a lower temperature with cuprous thiocyanate. One of the products is phenyl sulfide.¹¹¹⁵ 2-Hydroxy- α -naphthyl chloride gives a high yield of 2,2'-dihydroxynaphthyl sulfide under far milder conditions.¹¹⁰³ Phenyl iodide is considerably more reactive than phenyl chloride. Phenyl sulfide is formed when it is heated with a phenyl mercaptide: ^{149, 867, 1299}



This can be used for unsymmetrical aryl sulfides also.

Lime, sulfur, and phenyl chloride, heated together, give diphenyl sulfide.^{820, 821b} Distillation of *o*-tolyl selenocyanate gives di-*o*-tolyl selenide.^{88a}

Sulfides are among the products when a Grignard reagent is treated with sulfur¹³⁸⁷ or with sulfur chloride.⁴¹⁷ Selenides are obtained similarly.^{1000, 1017, 1235} These cannot be classed as methods of preparation.

The addition of bromine to a mixture of benzene, aluminum chloride, and sulfur is said to give a good yield of phenyl sulfide. Hydrogen bromide is evolved. It may be assumed that the bromine combines with the sulfur and that the sulfur bromide then reacts with the benzene under the catalytic influence of the aluminum chloride.^{131a, 302} With this catalyst, benzene and sulfur chloride give diphenyl sulfide along with more or less thianthrene.^{133, 134, 550} An aluminum-mercury couple may serve as catalyst.³¹⁷ A selenium halide reacts similarly to give diphenyl selenide.^{269, 725, 794, 814} Aluminum chloride converts ortho- and para-tolyl mercaptans to the sulfides.³⁶²

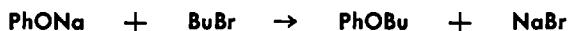
Benzene and sulfur in the presence of aluminum chloride give diphenyl sulfide along with thianthrene.^{131a, 373a} Benzene ^{6a} and naphthalene ^{6b, 1190b} react with sulfur chloride in the presence of zinc chloride,^{6a, 1146, 1190b} or aluminum chloride.^{1295.5} Naphthyl sulfides are formed when naphthalene, sulfur chloride, and aluminum chloride are heated together.¹³⁵⁸

Alkyl selenides are obtained from alkyl halides and selenium sulfide.^{120b} Benzyl selenides are prepared similarly.^{461, 653} Aryl selenides can be made from the diazonium salts.^{757c, 763, 1148, 1149} They can also be obtained from selenium tetrabromide and mercury salts: ^{757b}



UNSYMMETRICAL ALKYL SULFIDES

Williamson's synthesis of a mixed ether from a sodium alcoholate and an alkyl halide was epoch-making in that it fixed the atomic weight of oxygen at 16, but as a preparation method, it is poor. The sodium alcoholate is so readily hydrolyzed that the presence of water must be avoided and it is so alkaline that it abstracts hydrohalide acid from the alkyl halide. It is difficult to get a yield of better than 30 or 40% of a mixed ether by this method. However, excellent yields of phenyl ethers from sodium phenate and an alkyl halide, are obtained even in aqueous solution:



The sodium phenate is less basic and less hydrolyzed than the alcoholate.

In contrast to the Williamson synthesis of ethers, the reaction of an alkyl halide with a sodium mercaptide gives an almost quantitative yield of sulfide:



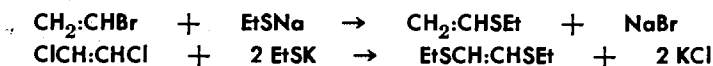
This is true in spite of the fact that the sodium mercaptide is hydrolyzed almost as completely as the corresponding alcoholate. The reason for this is to be found in the extremely high reaction rates when bivalent sulfur is involved. When an alkyl bromide is added to an alcohol solution of, say, potassium butyl mercaptide, and the mixture warmed even slightly, the precipitation of potassium bromide can be observed within a few seconds. The production of an alkyl sulfide, simple or mixed, is one of the simplest organic preparations. Although alcohols are much more available than mercaptans, mixed aliphatic sulfides are far better known than mixed ethers. In the preparation of a solid mixed alkyl sulfide, it is practicable to use as little as 0.5 g. of cetyl bromide, 0.33 milligram mole, with the corresponding amount of a mercaptan and alkali, and get an almost quantitative yield of the sulfide.

As to the actual operations in the preparation of an unsymmetrical sulfide what was said about making symmetrical sulfides applies. In this case, there are three reactants, alkyl halide, mercaptan, and alkali. It is customary to put the mercaptan

and alkali in together and add the alkyl halide at such a rate that the reaction, which is exothermic, is kept under control. Attention should be called to the fact that a mercaptan, in alkaline solution, is oxidised rapidly by air to the disulfide. The rate of oxidation depends on the concentration of the alkali and the temperature; the amount of oxidation depends on the time and manner of exposure. There should be a minimum of delay after the mixing of the alkali and mercaptan before the start of the halide addition. A better way of operating is to mix the mercaptan and alkyl halide and run in the alkali, in concentrated solution, at a proper rate. If alcohol is the reaction medium, the alkali halide is precipitated and may be filtered off. The alkyl sulfide may be isolated by distilling off the alcohol or thrown out by the addition of water. Volatilities and solubilities have to be considered in planning operations.

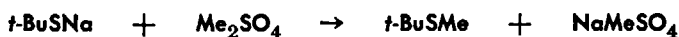
A mixed sulfide can be prepared from two pairs of starting materials. Thus, for methyl butyl sulfide, MeSBu, these may be methyl mercaptan and butyl bromide or butyl mercaptan and dimethyl sulfate. The choice would depend on the availability and convenience of handling. In this case, the use of the too volatile methyl mercaptan, boiling at 7.6°, would be avoided. As explained before, it is not necessary to isolate the mercaptan when it is prepared by the thiourea method.

Unsymmetrical sulfides have been prepared in great number and variety from alkyl halides and metal mercaptides.^{162, 193, 198, 209, 228.5, 306, 392, 552, 565a, 567, 597, 696, 740, 742, 773, 785, 832b, 915c, 950, 973.5, 1040, 1084, 1140, 1183, 1294.5, 1296, 1334} Dialkyl sulfates and the esters of *p*-toluene sulfonic acid are excellent alkylating agents.^{116, 377a, 377b, 484, 863a, 1180, 1194, 1413} The monosulfates and phosphates of the higher alcohols may be employed.⁵⁶⁸ Even vinyl bromide⁷⁹³ and 1,2-dichloroethylene^{338, 457, 463} react regularly:

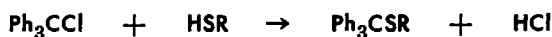


Terpene^{960a} and heterocyclic⁷⁹⁸ halides react satisfactorily with alkaline mercaptides. Conversely, terpene^{158c} and heterocyclic^{244b, 488, 693a} mercaptans are readily alkylated. This is true of 2-thiophenthio⁸⁹⁴ and 3-thiophenthio²⁰⁹. Mercaptoimidazoles, in alkaline solution, are alkylated by alkyl halides in the usual way.^{844, 1237} A variety of these sulfides has been so prepared.¹¹²

Methyl-2-mercapto-4-imidazolecarboxylate, when heated to 200°, passes into 2-methylmercaptoimidazole with loss of carbon dioxide.⁶⁵⁹ Tertiary mercaptans react in the same way as other mercaptans though somewhat tardily:¹⁰⁹⁵



Triphenylmethyl chloride is peculiar in that it reacts with mercaptans in ether solution without any alkali being present:^{113, 753a, 1328a}



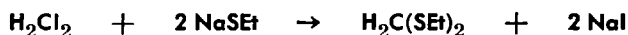
The same sulfides can be made from triphenylmethyl mercaptan:^{889, 1328b}



With sodium sulfide, the yield of triphenylmethyl sulfide, $(\text{Ph}_3\text{C})_2\text{S}$, is poor.^{1328b}

Sulfides are formed from alkyl chlorides and mercaptans in the presence of Friedel-Crafts catalysts, such as aluminum chloride, boron trifluoride, and stannic chloride.³⁸⁷

Multiple sulfides are produced when a polyhalide reacts with a mercaptide. Thus methylene iodide and a sodium mercaptide give thioformals:^{455a, 700a, 1240}

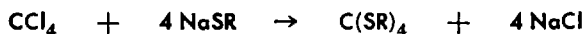


The thioformals are also prepared from formaldehyde and the mercaptans.⁷⁷⁰ Ethylene,^{403, 1203.5, 1345, 1366b} trimethylene,¹⁰⁷⁰ octamethylene, decamethylene, and dodecamethylene^{190a} halides react with two molecules of mercaptides to produce *bis*-sulfides, $\text{RS}(\text{CH}_2)_n\text{SR}$. Similar compounds can be made, the other way around, by the reaction of alkyl halides on ethylene mercaptan^{863a} and on 2,2'-dimercaptoethyl sulfide, $\text{HSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SH}$.^{22, 863a} From trimethylene mercaptan the *bis*-sulfides, $\text{RSCH}_2\text{CH}_2\text{CH}_2\text{SR}$,^{42, 1239} have been prepared. This mercaptan and trimethylene bromide gave a polymer, $(\text{CH}_2\text{CH}_2\text{CH}_2\text{S})_x$.⁴² A variety of polymers of this sort were obtained as by-products in making cyclic sulfides from dimercaptans, $\text{HS}(\text{CH}_2)_n\text{SH}$, and dihalides, $\text{Br}(\text{CH}_2)_n\text{Br}$.^{878, 1304} In a study of the melting points of sulfur compounds, the series $\text{RSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SR}$ was completed up to $\text{R} = \text{octadecyl}$. A great many *bis*-sulfides, $\text{RS}(\text{CH}_2)_n\text{SR}$, in which $n = 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12$, and 18 and

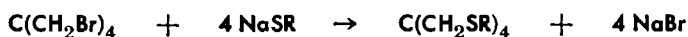
R was from hexyl to octadecyl, have been prepared from the dimercaptans.¹⁰⁸⁰

The tris-sulfide $\text{MeSCH}_2\text{CH}(\text{SMe})\text{CH}_2\text{SMe}$, has been prepared from 1,2,3-trimercaptopropane and dimethyl sulfate.¹⁰⁹⁶

Carbon tetrachloride was supposed to react according to the equation: ^{700b}



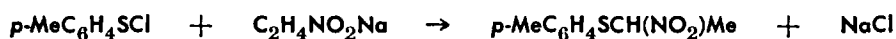
Later investigation showed that the product was the trithioformic ester, $\text{HC}(\text{SR})_3$, mixed with some of the disulfide, $\text{RS}\cdot\text{SR}$.^{55a, 55b, 55c} The true sulfides, $\text{C}(\text{SR})_4$, aliphatic^{54, 55a, 55b, 55c} as well as aromatic^{28, 55a, 55b, 55c} have been obtained in a roundabout way. The tetrabromide, $\text{C}(\text{CH}_2\text{Br})_4$ from pentaerythritol reacts regularly with mercaptides:^{51a, 293}



The orthoformic esters, $\text{HC}(\text{SR})_3$, are usually made from ethyl formate and a mercaptan in the presence of hydrochloric acid.^{602a} These will be discussed in the volume on thioacids.

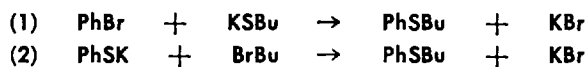
Though it is hardly a preparation method, the formation of alkyl sulfides by way of a sulfonium compound is to be noted.¹⁰⁵⁸ This will be treated more fully under cyclic sulfides.

A peculiar way of making a sulfide is the reaction of a sulphenyl halide with the sodium salt of a nitroparaffin: ⁶⁸⁷



UNSYMMETRICAL ARYL AND ARYL-ALKYL SULFIDES

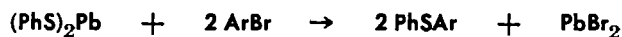
If aryl halides were as reactive as their alkyl analogs a mixed aryl-alkyl halide could be prepared equally well in two ways:



However, aryl halides are unreactive and reaction (1) does not take place under ordinary conditions. PhBr is bromobenzene rather than phenyl bromide. Its reactivity can be enhanced by the presence of a nitro group. It is quite different with reaction (2). PhSH is phenyl mercaptan and behaves like an aliphatic mercaptan. The removal of the sulfur atom from its place in the benzene ring is not involved. This reaction goes 600 to 1000 times as fast as the corresponding reaction with potassium phenate.¹⁰⁴⁶

The reaction rate is influenced by substituents in the β -position in the alkyl halide.^{582,5}

An aromatic bromide does react with a lead mercaptide at a high temperature: 169a, 169b, 171, 227, 724



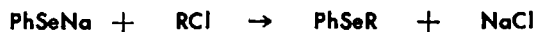
As mentioned under symmetrical sulfides, 2-nitro-, 4-nitro-, and 2,4-dinitro-phenyl chlorides react satisfactorily with metal mercaptides.^{486, 498a, 498b, 588, 590a, 709, 749, 1230, 1263, 1337, 1356} A nitro group is replaced when other nitro groups are in the 2,4-positions.^{498a, 498b} 2,4-Dinitrophenyl chloride may react with a nascent mercaptan.¹⁰³² When it is heated with benzylthiuronium chloride and alkali added, benzyl 2,4-dinitrophenyl sulfide is formed.⁶³ This chloride has been recommended for the identification of mercaptans. With sodium mercaptides, it gives the sulfides, 2,4-(NO₂)₂C₆H₃SR, which have satisfactory melting points. They may be oxidised to the sulfones.^{105, 166, 434} A number of these have been listed in Chapter 2 of Volume I, under the identification of mercaptans. In 3,4,5-triiodonitrobenzene, the iodine in the 4-position is activated.^{126, 649, 1136}

A diazonium chloride reacts with a mercaptide: 508, 509, 566, 658, 678, 1113.5, 1211, 1347



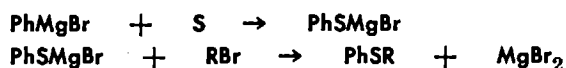
Numerous aryl-alkyl sulfides have been prepared from thiophenol and its substitution products^{44a, 170, 190a, 272, 440, 490, 626, 707, 977b, 1265, 1337, 1356, 1364, 1407a, 1410, 1413} and from the thionaphthols.^{707, 727} The alkyl biphenyl sulfides, *p*-PhC₆H₄SR, from hexyl to octadecyl have been described. In a study of the melting points of the members of series, a large number of aryl-alkyl sulfides have been synthesized from thiophenol, its *p*-methyl- and *p*-bromo- derivatives and from β -thionaphthol with the higher alkyl bromides up to octadecyl and eicosyl.¹⁰⁸⁰ Methyl *o*-cresyl sulfide has been described.⁴⁴⁵ Benzyl nitrate serves as an alkylating agent.²⁵⁵

Sodium selenomercaptan reacts regularly with an alkyl halide: 439

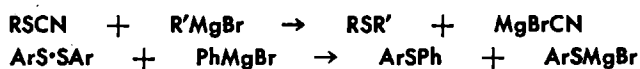


When a thiophenol, or selenophenol, is prepared by the Grignard reaction, it is not necessary to isolate it before reacting it

with an alkyl halide ^{489, 1261a, 1261b, 1261c, 1261d} or other alkylating agent: ^{89, 400, 485, 491, 492, 494, 571, 754, 1346}



A Grignard reagent cleaves an alkyl thiocyanate or a disulfide: ^{3, 233b}



A sulfide is one of the products in each case. A diselenide is similarly cleaved.²⁵¹

Unlike phenols, aryl mercaptans react with dialkyl sulfates to form sulfides in the absence of alkali as well as in its presence.⁹⁶

p-Bromothiophenol and chlorobenzene react, in the presence of aluminum bromide, with the elimination of hydrogen bromide instead of the chloride. The product is *p*-chlorophenyl phenyl sulfide, *p*-ClC₆H₄SPh. Similar reactions take place with benzene and toluene. Mechanisms have been suggested for the rather complicated reactions which are involved.¹³⁷³

SULFIDES FROM THE ADDITION OF MERCAPTANS TO UNSATURATES

The simplest possible formation of a sulfide is the direct addition of a mercaptan to an unsaturate:



Examples of such addition have been known for some time but have been considered special cases. Only recently has the generality of this reaction been realized and the conditions governing it determined. Now it takes its place as an important method for the synthesis of sulfides which are being reported so rapidly that a catalog of them would be out of date before it could be typed. This subject has been reviewed.⁷¹⁰ The addition of mercaptans to unsaturates has been mentioned in Chapter 2 of Volume I, among the reactions of mercaptans.

This addition was observed by Posner¹⁰¹⁹ and others^{36, 1122a} but they used thiophenol, *p*-thiocresol, and benzyl mercaptan, which are much more reactive than the simple aliphatic mercaptans. The double bonds in their unsaturates were activated by the proximity to carbonyl or phenyl groups. They found that the

addition takes place contrary to Markownikow's rule. By the addition of thiophenol to styrene, β -phenylmercapto-ethylbenzene is produced:



It was noted that "the rate of addition is greatly dependent on the intensity of the illumination and the increased rate produced by sunlight continues for some time after the reaction vessel is returned to diffused light."³⁶ The effect of light will be taken up again later on in this chapter. The addition of *p*-thiocresol to divinylacetylene was used as a proof of its structure.²⁵⁷ Cyclohexene and methyl mercaptan combine in the presence of ultra-violet light.⁷⁷ The addition of ethyl mercaptan to cetene takes place under the same conditions, but without the illumination there is no reaction.^{948b} Butyl crotyl sulfide is produced by the addition of butyl mercaptan to butadiene.⁷⁹⁹

In a study of the addition of mercaptans to simple unsaturates, it was found that the abnormal addition is due to the presence of traces of peroxides. Only minute amounts, such as are normally present in most organic compounds, handled and stored without special precautions, are sufficient. If special pains are taken to eliminate these, the addition goes according to Markownikow's rule, if at all.⁶⁶⁰ It was found that thioglycolic acid reacts quickly with styrene, in the presence of ascaridole, to form $\text{PhCH}_2\text{CH}_2\text{SCH}_2\text{CO}_2\text{H}$, but that no addition takes place in the presence of hydroquinone.⁶⁸⁴ The peroxide effect in the addition of mercaptans to unsaturates has been covered in a review by Mayo and Walling.⁸⁷⁴ The structures of a number of the addition products have been rigorously established by synthesis by other methods and some errors found in Posner's work.^{647, 648} Methyl and benzyl mercaptans have been added to several unsaturates in the presence of ascaridole.²²⁰

Mercaptans may be added to unsaturates in the presence of oxygen.^{947a} Thus, propyl mercaptan and styrene give propyl β -phenylethyl sulfide. A free-radical mechanism has been proposed for this reaction.⁶⁸³ Sulfides and sulfones are said to be formed when oxygen is introduced, at 200°, into a mixture of olefins and mercaptans.⁵⁴⁴

Mercaptans differ greatly in their activity in addition reactions. The aromatic mercaptans, thiophenol and *p*-thiocresol, are very

active; benzyl mercaptan seems to be slightly less so. These mercaptans combine with many unsaturates spontaneously. At least this is true of the compounds which have been exposed to air. The simple aliphatic mercaptans are much less reactive and may require heat, or ultraviolet light, and the assistance of catalysts, such as peroxides or alkali. Ethylene dimercaptan is very reactive, even more so than thiophenol. This appears to be due to the influence of the sulfur atom in the beta position with regard to the $-SH$ group. Mercaptoethanol, $HSCH_2CH_2OH$, and thioglycolic acid are extremely reactive. A quantitative study of the reactivity of various mercaptans is most desirable. The task is beset with serious difficulties since the reactivity is enhanced so greatly by fortuitous traces of peroxides and depressed or inhibited by traces of impurities, such as sulfur and copper. As will appear in the examples given later, all sorts of catalysts are recommended.

There are great differences among the unsaturates in their susceptibility to addition. It is known that an open vinyl group $-CH:CH_2$ is favorable to addition of a mercaptan as it is to polymerization which also involves addition. Thus β -pinene, in which the double bond is between carbons 1 and 7, is far more reactive than α -pinene in which it is between carbons 1 and 2.¹⁰⁷⁹

The addition of thioglycolic acid to unsaturates will be discussed later as a method for the synthesis of sulfide acids. It has been mentioned in Chapter 5 of Volume I as a reaction of that acid. Thiophenol adds spontaneously to many unsaturates, to heptene-1, undecene-1, dodecene-1, diallyl, ethyl-vinyl ether, divinyl ether, and to methyl acrylate. On mixing 11 cc. of butadiene-mono-epoxide and 7 cc. thiophenol, the temperature went up 22° in 8 minutes. A mixture of thiophenol and undecylenic acid solidified in 2 days. The recrystallized product melted at 82.5° . The acid number was found to be 294.5 and 294.8; calculated for $PhS(CH_2)_{10}CO_2H$, 294.4.¹⁰⁷⁹ 3-Thiophenethiol adds spontaneously to styrene.²⁴² When ethylene mercaptan is poured into acrylonitrile, the addition is immediate and so much heat is evolved that the mixture may be ejected from the flask unless the one is poured into the other with strong cooling. This mercaptan reacts slowly but completely with allyl alcohol, with terpenes, and with cyclohexene. β,β' -Dimercaptoethyl sulfide, $HSCH_2\cdot CH_2\cdot SCH_2\cdot CH_2SH$, is also an active mercaptan. Its mixture with two

equivalents of allyl alcohol shows no immediate heat effect but, in about 20 minutes, the flask is too hot to hold in the hand.¹⁰⁷⁹

The addition of a mercaptan to an unsaturate is believed to be a chain reaction activated by a free radical.^{683,5} Ultraviolet light is effective.⁵²⁷ The light must act on something capable of giving a free radical. This is illustrated by a study of the addition of methyl mercaptan to allyl alcohol:



With light, mercuric methyl mercaptide, $(\text{MeS})_2\text{Hg}$, in the reaction mixture, and oxygen over it, the yield was 93%. With light, but without the mercury salt and oxygen, there was no reaction; with the salt and oxygen 22%; with the salt and light 33%; and with oxygen and light 61%.⁶⁶⁶ Methyl mercaptan has been added to acrylic acid, acrolein, maleic acid, and styrene.⁶⁶⁷ Dodecyl mercaptan has been added to mesityl oxide and to methyl acrylate.¹²³⁰ Trimethylene- and hexamethylene-dimercaptans are less reactive. They add to two molecules of cyclohexene with the aid of ultraviolet light.^{336d} Some forty mercaptans have been added to safrole and isosafrole in the presence of ascaridole. The products were tested for insecticidal properties.^{1036, 1251, 1252} *t*-Butyl mercaptan is said to be added to olefins, under pressure at 30 to 200°, in the presence of nickel, iron, or cobalt sulfides.^{12, 13a, 594, 949b} Peroxides, with heavy metal salts of strong acids, are recommended as catalysts.^{593, 947a} *p*-Thiocresol and β -thionaphthol have been added to a number of unsaturates. The catalyst was 0.01 mole of piperidine added to the mercaptan dissolved in 2.5 times its weight of absolute alcohol.⁸²³ Thiophenol and the thiocresols have been added to 2,5-dimethyl-1,5-hexadiene.²²⁷ Dithioresorcinol has been added to two molecules of each of a number of unsaturates.⁴³¹ Cyclohexene and 2-methylcyclohexene unite with thiophenol when irradiated. The same is true of cyclohexene with *i*-amyl mercaptan.^{336a, 336c} Thiophenol, benzyl mercaptan, and aliphatic mercaptans add to cyclohexene^{1349a} and to dicyclopentadiene.²²⁸

Thiophenol^{632a, 637a, 637c, 1201, 1349a} and other mercaptans^{629c, 632a, 637a, 689, 1035.5, 1079, 1086b, 1087b} unite with acetylene at elevated temperatures and in the presence of basic catalysts:

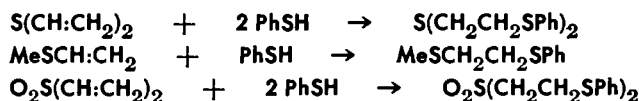


A second molecule may be taken up:



When acetylene and hydrogen sulfide are passed through glycerol, containing a sulfide catalyst, ethyl vinyl sulfide is obtained along with ethyl mercaptan and $\text{EtSCH}_2\text{CH}_2\text{SEt}$.^{633a, 1086c}

The addition of thiophenol to divinyl sulfide,¹⁰ methyl vinyl sulfide,²⁸¹ and to divinyl sulfone^{735b} takes place spontaneously:



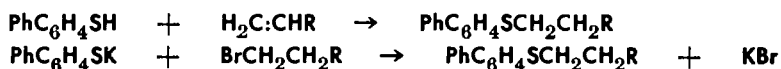
Methyl mercaptan adds to propenyl phenyl sulfone, $\text{PhSO}_2\text{CH:CHMe}$, in the presence of trimethylamine.^{860.5}

It is claimed that useful products are obtained by the addition of mercaptans to vinyl ketones,^{1118.5} vinyl thioethers,^{629d} RSCH:CH_2 , vinyl sulfones,⁶³⁸ $\text{RSO}_2\text{CH:CH}_2$, or N-vinyl pyrolic compounds,^{632b} or a vinyl-phosphonic ester, $\text{CH}_2\text{:CHPO(OEt)}_2$.¹²⁷¹

Addition to acrylonitrile takes place readily.^{86, 823, 1179} Various mercaptans have been added to it in the presence of "Triton B" or an alkali. Thiophenol required no catalyst.⁶²⁵ The nitrile is added to an aqueous alkaline solution of the mercaptan. The exothermic reaction is controlled by cooling.⁵⁴⁵ 3-Thenyl mercaptan, $\text{C}_4\text{H}_9\text{SCH}_2\text{SH}$, adds to acrylonitrile in the presence of an alkaline mercaptide.^{693a} Piperidine has been used as a catalyst.^{1116, 1117} 2-Mercaptothiazole adds in the presence of a catalyst.³¹³

Methyl, ethyl, and benzyl mercaptans have been added to a number of unsaturates in the presence of "Triton B" or potassium carbonate. One mole of the mercaptan is dissolved in 200 cc. of a solvent with 10 g. of the catalyst. The unsaturate is added in portions with stirring.¹²⁵⁴

Products suitable for the identification of alkenes may be prepared by selecting mercaptans that are apt to give solid sulfides. The addition products of 4-mercaptodiphenyl with a number of α -alkenes have been made and compared with the sulfides prepared by the usual method:



The products were identical.⁷⁶⁴

Mercaptans can be added to α -nitroolefins under basic conditions.^{60, 266, 561, 562, 743}

A mercaptan and an olefin unite when heated to 225° in the

presence of selenium. Nothing is said as to which way the addition goes.¹³³⁹ Addition is said to take place in the presence of practically anhydrous phosphorus pentoxide.^{949a} The process may be made continuous by using hydrofluoric acid as a catalyst.^{1158b} Under acid conditions, the addition is probably according to Markownikow's rule. Mercaptans have been added to dihydropyran in the presence of hydrogen chloride.^{692, 693b} Boron trifluoride etherate,^{210a} boron trifluoride-hydrofluoric acid,^{93b, 955} aluminum chloride, and cuprous chloride^{93b} are catalysts. Activated montmorillonite is a catalyst for such addition.¹¹⁸¹

The formation of polymers by the addition of dimercaptans to diolefins has been investigated extensively by Marvel and associates. Molecular weights up to 60,000 have been attained.⁸⁵¹ Some lower polymers from hexamethylenedithiol are rubberlike.⁸⁵³ Various initiators have been used.⁸⁶⁰ Polymers can be obtained by exposing a mixture of a dimercaptan and a diolefin to ultraviolet light at an elevated temperature.^{316a} With the aid of ultraviolet light, polymers have been obtained with sufficiently high molecular weights to be cold-drawn.⁸⁵² A high polymer was obtained from hexamethylene dimercaptan and biallyl.^{855, 856.5} This was duplicated by causing the same mercaptan to react with hexamethylene bromide in alkaline solution.⁸⁵⁵ A similar polymer was made from this mercaptan and tetramethylene bromide.⁸⁵⁷ Hexamethylenedithiol and β -phenyl-vinyl ketone, $(\text{PhCH:CH})_2\text{CO}$, gave a polymer of 60,000 molecular weight.⁸⁵⁸ The formation of emulsion polymers from the dienes, butadiene and biallyl, with hexamethylene dimercaptan^{852, 855, 856, 858, 859} and with 4,4'-dimercaptodiphenyl ether⁸⁵⁴ has been studied extensively. A dithiol has been added to divinylacetylene in the presence of alkali.⁷⁴⁵ Ethylene mercaptan and divinyl ether unite spontaneously forming a hard white polymer.¹⁰⁷⁹ Polymers have been obtained from other dienes and dimercaptans.^{856, 910.5}

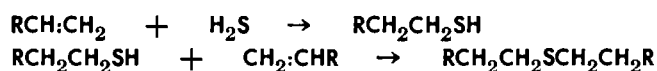
Allyl mercaptan, having a vinyl group at one end and a mercaptan at the other, adds to itself to form a linear polymer.^{196, 197} Cinnamyl mercaptan polymerizes on standing.¹⁹⁷

Low polymers, said to be suitable for lubricating oils, are made by the addition of hydrogen sulfide, or a mercaptan, to divinyl, or diallyl, ether. Various catalysts may be used.^{949e} A polyalkylene sulfide, $\text{R}(\text{SCHR}'\text{-CHR}'')_x\text{OH}$, results from the reaction of a tertiary mercaptan and an alkylene oxide in the presence of an acid catalyst.³⁵⁰

The sulfene chlorides, $p\text{-NO}_2\text{C}_6\text{H}_4\text{SCl}$ and $o\text{-NO}_2\text{C}_6\text{H}_4\text{SCl}$, add to unsaturates, such as cyclohexene and cyclopentene.^{688, 1307} This has been discussed in Chapter 3, Volume I. The products are chlorosulfides which will be treated in Chapter 4.

SULFIDES BY THE ADDITION OF HYDROGEN SULFIDE TO OLEFINS

Actually this is a case of a sulfide being formed by the addition of a mercaptan to an olefin. The addition of hydrogen sulfide to an unsaturate forms a mercaptan as has been stated in Chapter 1, Volume I, on mercaptans. The mercaptan so formed finds itself in the presence of some unreacted unsaturate and union takes place. There are two reactions:



It is to be expected that the two reactions will have quite different rates according to the structure of the unsaturates. The amounts of mercaptan and sulfide formed from several olefins are shown in Table 1.1.

TABLE 1.1
*Percentages of Mercaptans and Sulfides Formed
by Heating Olefins with Hydrogen Sulfide*

Olefin	Mercaptan	Sulfide
Ethylene	11	80
Propylene	7	90
Isobutylene	23	6
Cyclohexene	7	5

The ethyl and propyl mercaptans add to the unsaturate almost as rapidly as they are formed, that is, the rate of the second reaction is rapid compared to that of the first. *t*-Butyl and cyclohexyl mercaptans do not add so rapidly.⁶⁶⁰ These statements hold for one set of conditions only. Far better ways of effecting the additions have since been found.

The important observation was made by Vaughan and Rust that the addition of hydrogen sulfide and of mercaptans to unsaturates is enormously accelerated by ultraviolet light, particularly in the presence of substances which dissociate under irradi-

ation. Butene-1 and hydrogen sulfide in a sealed tube, irradiated 4 minutes at 0° , combined to the extent of 80%. The product was about 80% butyl mercaptan and the rest butyl sulfide. With propylene and hydrogen sulfide, the conversion was 75% in the presence of acetone but only 4% in its absence under the same illumination.^{401, 1124, 1318, 1319} It is to be noted that the relative proportions of mercaptan and sulfide are very different from the figures quoted before. The combination of light and catalyst is particularly favorable to the primary reaction, that is, the addition of hydrogen sulfide to the olefin. From cyclohexene, 1-methylcyclohexene, and dihydromyrcene, with hydrogen sulfide, mercaptans and sulfides were obtained. The proportion of sulfide was greater with cyclohexene than with its 1-methyl derivative. The dihydromyrcene gave a cyclic sulfide. The addition was in the presence of ultraviolet light with acetone to furnish the free radicals.⁹³⁸ Cyclic sulfides were obtained from dihydromyrcene and geranolene under different conditions.¹¹⁷⁷ Certain azo compounds catalyze such addition.¹⁰⁰³

When ethylene, propylene, butene-1, or *i*-butylene and hydrogen sulfide are passed over silica gel at 650 to 725° , mercaptans and sulfides are formed. These may go into thiophenes.^{833b, 834}

The addition of hydrogen sulfide to an unsaturate may serve as a method of preparing mercaptans or their derivatives on a commercial scale. A base is recommended as a catalyst.^{633b, 636, 638, 682} An olefin is treated with hydrogen sulfide under pressure, at 35 to 300° in the presence of a water-binding agent, such as acetanhydride. A metal sulfide may serve as a catalyst.^{948c, 1370, 1371} The alkene may be treated with an acid, such as sulfuric, and then with hydrogen sulfide.^{648, 756} The olefin and hydrogen sulfide may be absorbed in concentrated sulfuric acid and the mixture diluted with water.¹¹²⁵ Hydrogen sulfide is added to abietyl compounds, or to other unsaturates of high molecular weight, in the presence of a catalyst, either an acid or a base, under nonoxidising conditions.^{155, 960d} Compounds, some of which are sulfides, can be obtained by subjecting unsaturates to the simultaneous action of hydrogen sulfide and sulfur in the presence of a base.^{11, 1018}

Ethylene heated with a solution of sodium ethylate in ethanol, while hydrogen sulfide is passed in, gives ethyl sulfide.⁶³⁶ Thioethers containing the pyrrol radical can be prepared by the addi-

tion of hydrogen sulfide or a mercaptan to N-vinyl-pyrrole.^{630a, 632b, 637b, 1087a} Boron trifluoride-phosphoric acid and boron trifluoride-water are suggested as catalysts.^{1158a} Sulfides are formed along with mercaptans when hydrogen sulfide is caused to react with olefins in the presence of Friedel-Crafts catalysts.^{84, 93a, 95, 1100, 1216a, 1216b}

An olefin, hydrogen sulfide, and hydrogen, at 200° and 1000 lb. pressure in the presence of cobalt trisulfide catalyst, give a mixture of mercaptan and sulfide. Sulfur, which combines with a part of the hydrogen, may be used instead of hydrogen sulfide.¹⁵

When ethylene is continuously supplied to hydrogen sulfide at 700 to 900 atmospheres of pressure, in the presence of dilute hydrochloric acid, the addition does not stop at ethyl mercaptan and ethyl sulfide. Telomerization takes place with the formation of products of the general formula $H(CH_2CH_2)_nSEt$. Butyl ethyl sulfide is the first of a series which may extend to much higher molecular weights.^{384b, 546}

Hydrogen sulfide and nitro olefins combine in the presence of sodium methylate to give nitroalkyl sulfides.⁵⁶²

In the acid-catalyzed reactions the addition is usually according to Markownikow's rule.

The addition of hydrogen sulfide to acetylene should give vinyl mercaptan as the primary product:



This would isomerize into thioacetaldehyde which would immediately trimerize to trithioacetaldehyde. When the two gases are passed into a solvent, such as dioxane containing potassium hydrosulfide, ethyl mercaptan, ethyl-vinyl sulfide and bis(ethyl-mercapto) ethane may be by-products.^{633a} See the chapter on thioaldehydes in Volume III.

SULFIDES FROM SULFUR AND HYDROCARBONS

This is starting a step farther back. The hydrogen sulfide, that is to be added to the olefin to make the mercaptan, which is to be added to the olefin to form the sulfide, has to be produced. The olefin may come from the reaction of the sulfur with a paraffin. These are three consecutive reactions:

- (1) Sulfur with the hydrocarbon.
- (2) Addition of hydrogen sulfide to the olefin.

(3) Addition of the mercaptan to the olefin.

At first sight, it seems curious that a sulfide should be produced regardless of the starting materials. The explanation is, however, simple enough. When a hydrocarbon is heated with sulfur, reactions of various kinds take place. Hydrogen sulfide is always produced and some of it combines with the original hydrocarbon or with one of its decomposition products to form a mercaptan. Thus cyclohexene and sulfur, heated together at 150°, give cyclohexyl mercaptan and cyclohexyl sulfide.⁸⁹¹ Ethyl sulfide is produced when:

(1) Ethylene is heated with ethyl mercaptan.

(2) Ethylene is heated with hydrogen sulfide.

(3) Ethylene is heated with sulfur.

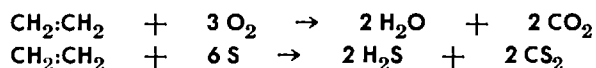
An end product is ethyl sulfide in each case. In addition, there may be some ethylene sulfide also.⁶⁶⁰

REACTIONS OF SULFUR WITH VARIOUS HYDROCARBONS

This is taken up here since sulfides are usually among the products. It is, however, a large and complicated subject and can be given only token treatment here. Reference must be made to reviews that have appeared.^{448b, 719, 988, 1258a, 1359} 2-Methylbutene-2 and sulfur, at 170°, give 3-methylbuten-2-yl sulfide and mercaptan.²¹⁴

As shown in the comparatively simple case of ethylene and sulfur, there are several reactions and a number of products. With higher hydrocarbons there are the same reactions: dehydrogenation and addition of hydrogen sulfide and mercaptans to unsaturates, but there are more possible ways in which these reactions can take place. In any case the results will depend on the conditions, proportion of sulfur, and time and temperature of heating. The hydrocarbon may undergo dehydrogenation, cracking, and polymerization independent of the sulfur and then the sulfur may react with the products so formed.

At higher temperatures, sulfur reacts with hydrocarbons to form hydrogen sulfide and carbon disulfide which are analogous to water and carbon dioxide, the usual combustion products.¹³¹⁶



When ethylene is bubbled through sulfur at 400°, about 65% of the carbon appears as carbon disulfide.¹⁰⁷⁹ At 280 to 320°, sulfur

and a paraffin or cellulose give hydrogen sulfide.¹¹⁹¹ Heating a heavy fuel oil with sulfur is a convenient and economical way of generating hydrogen sulfide.²⁰⁸ Carbon disulfide is formed in the cracking of a high sulfur crude oil.⁶⁰⁴ Methane and sulfur give carbon disulfide and hydrogen sulfide.⁹²⁹ The manufacture of carbon disulfide by this method, in the presence of catalysts, has been proposed.^{367, 1276a, 1278} The production of carbon disulfide from hydrogen sulfide and hydrocarbons at 800 to 1000° has been investigated.¹⁰⁴⁸

When various hydrocarbons are heated strongly with excess sulfur, black insoluble infusible residues are obtained. These have the approximate composition, C_2S .^{980, 1273} This suggests a polymeric sulfur acetylide, $(C:CS)_n$. With less drastic treatment, colored insoluble residues, which still contain hydrogen, are obtained.^{847, 980} These are soluble in sodium sulfide and may be used as dyes.^{788, 969, 970} Soluble black dyes are obtained by boiling furfural with aqueous sodium polysulfide. Structures have been proposed for these, but not proved.⁸³⁷

Much attention has been given to the reactions of sulfur with the terpenes, which are readily available and reactive.^{154, 474, 603, 713, 720, 843, 848, 885, 960b, 1026, 1027, 1127, 1143, 1173, 1215d, 1351} Under controlled conditions, a fairly definite sulfur compound, $C_{10}H_{16}S$, or $C_{10}H_{18}S$, is obtained by heating terpenes with sulfur. Diverse terpenes and terpene alcohols give products of similar characteristics. A preparation from Russian turpentine combined with methyl iodide to give $C_{10}H_{16}SMeI$, a sulfonium salt from which a strong base is obtained by treatment with silver oxide. With auric chloride, an oil-soluble gold compound is obtained. This is more like an aurous mercaptide in that it does not appear to contain any halogen. The constitution has not been determined. This compound has been used extensively in ceramics for depositing gold decorations on china.^{146, 229, 230, 651, 930, 931, 1391} This will come up again in the chapter on cyclic sulfides in Volume III. Sulfurized terpenes are claimed as cutting oils.^{388, 572, 669, 670, 711, 972, 1023}

More or less *p*-cymene may be produced by heating terpenes with sulfur.^{720, 1127, 1143}

Various other hydrocarbons have been heated with sulfur and the products isolated.^{32, 158a, 200, 211, 214, 283, 409, 447, 500, 629b, 845} Only a few examples will be noted. When naphthenes are heated with sulfur, benzene homologs are formed along with high-boiling

sulfur compounds.⁸⁴⁵ Cyclohexane and sulfur may give cyclohexyl mercaptan^{158a} or benzene, thiophenol, and diphenyl sulfide.⁴⁴⁷ Hexene gave $C_6H_{12}S$, $(C_6H_{12})_2S$, $C_{18}H_{34}S_2$, and $C_{24}H_{40}S_3$.⁴⁴⁷ 2-Cyclohexylbutene gave *s*-butylbenzene at 200° and cyclohexylbenzene and diphenyl at 250°. ²¹¹ Cyclohexene, below 150°, gave a series of saturated and unsaturated sulfides and polysulfides, $C_{12}H_{20}S$, $C_{12}H_{22}S$, $C_{12}H_{20}S_2$, $C_{12}H_{22}S_2$, etc.⁴⁰⁹ Thiophene and alkylthiophene may be obtained.⁴⁴⁷ 2,4-Diphenylthiophene is from sulfur and ethylbenzene.⁵⁰⁰ Zinc and vulcanization accelerators favor the reaction of sulfur with hydrocarbons.^{26, 27} Benzaldehyde, diphenylmethane, and benzyl and benzhydryl ethers go into tetraphenylthiophene. The same is true of dibenzyl and stilbene.^{1259, 1260} A good yield of benzothiophene results when hydrogen sulfide and styrene are passed over a catalyst.^{538, 908}

Alkyl sulfides, along with mercaptans, are said to be formed when methane, ethane, propane, and butane, mixed with sulfur vapor, are passed over various oxide or sulfide catalysts.^{1276b}

The formation of cyclic sulfides from sulfur and olefins is treated in the chapter on cyclic sulfides in Volume III.

Diverse products are obtained according to conditions. Olefins bubbled through molten sulfur or dispersed in a solution of sulfur at 160°, in the presence of a catalyst, give a mixture of low and high molecular weight organic sulfur compounds.^{382, 383, 1361} Carbon disulfide, alkyl sulfides, disulfides, and mercaptans may be formed.^{1276b} When olefins of two to four carbon atoms are brought into contact with sulfur at 170 to 250° at pressures up to 1000 atmospheres, sulfides and polysulfides are formed. Hydrogenation of the products from ethylene gives $EtSH$, $HSCH_2CH_2SH$, $EtSCH_2CH_2SH$, and $EtSCH_2CH_2SEt$, and similar derivatives.^{750, 1190a}

Ethylene and acetylene and sulfur at 300° give thiophene, carbon disulfide, hydrogen sulfide, and carbon.⁸⁹⁵ Small yields of alkyl thiophenes are obtained by heating sulfur in a sealed tube with octane^{448a} or heptane.⁶² Crude butadiene, bubbled through sulfur at 320 to 420°, yields 6% of thiophene.¹¹⁷⁵ 3-Phenylthiophene,^{212a, 215} 3-methyl-4-phenylthiophene,^{212a, 213} 2-methyl-4-phenylthiophene, and 2-methyl-3-phenylthiophene^{212b} are obtained when the appropriate substituted styrenes are heated with sulfur. β -Propylstyrene and β -isopropylstyrene and sulfur give 2-phenyl-5-methylthiophene and 2-phenyl-4-methylthiophene,

respectively, when heated with sulfur.¹³²⁹ Substitution products of stilbene go into the corresponding thionessal derivatives.⁷²²

Thiophene is one of the pyrolysis products of high-sulfur bituminous oils, the ichthyol oils.¹¹⁴¹

ACETYLENE AND SULFUR

When acetylene is passed into heated sulfur, the chief condensation product is thiophthene,^{252, 319} which bears the same relation to thiophene as naphthalene does to benzene. Others are thiophene,^{204, 286, 892, 1300} dimethylthiophene, thionaphthene, and tars.⁸⁹² Under different conditions thiophene may predominate and thiophthene take second place.⁹⁸³ Acetylene and sulfur at 290 to 390° give thiophene, thiophenol, and thiophthene.¹¹¹ At 650°, 83% of the sulfur goes to carbon disulfide and 5% to thiophene.⁹⁸³ Hydrogen sulfide may be substituted for the sulfur without much alteration of the products.^{30, 31, 286, 983, 1238} Hydrogen sulfide and carbon disulfide are always present in the reaction zone. Acetylene and hydrogen sulfide, over bauxite at 320°, give thiophene.^{286, 1238} Various catalysts are recommended for this reaction.^{30, 31, 631a}

Instead of sulfur, pyrites or some other substance that yields sulfur may be used.^{631a, 1224} Over pyrites at 280 to 310°, acetylene is converted to thiophene,¹²²³ isoprene to 3-methylthiophene, and 2,3-dimethylbutadiene to 3,4-dimethylthiophene.¹²²² When acetylene and carbon disulfide are passed through a tube at 700°, 10% of the condensate is thiophene.²⁰⁴

Recently thiophene has been manufactured on a considerable scale by passing butane and sulfur vapor through a hot zone. It is believed that the butane is first dehydrogenated to butene and butadiene. 3-Thiophenethiol and high molecular weight sulfur compounds are by-products.^{1053, 1054} 2-Methylthiophene and 3-methylthiophene are from pentane and isopentane, respectively.¹⁰⁵³ Thiophene is obtained from sulfur vapor and butadiene at 445°. ^{316b}

There are some articles and a number of patents on sulfurizing mineral oils and uses for the products.^{305, 402, 435, 576, 671, 708b, 783, 784, 830, 872, 886, 911b, 924, 940, 1215c}

Glycerol,²²² anise oil,⁸⁴² indophenols,³⁸ nicotine,¹⁷⁶ indole,^{1047, 1258b} carbazole,¹⁰⁴⁷ isophorone,⁶²⁴ and squalene^{130b} have been heated with sulfur and the products studied.

There have been many investigations of the action of sulfur

on unsaturated fatty acids and their glycerides. Various structural formulae have been proposed for the products, but none of these has been accepted generally.^{85, 397, 706, 797, 1051, 1131, 1213, 1363} The subject has been reviewed in part.⁷² This will be discussed again in Volume III when factice is considered. Sulfurized unsaturated acids and esters enter into a variety of products which are claimed to be useful.^{14, 33, 104, 321, 503, 523, 708a, 714, 781, 782, 802, 911a, 953, 954, 997, 1022, 1025, 1109, 1200, 1214, 1404}

ADDITION OF SULFUR CHLORIDE TO UNSATURATES

As will be shown in detail in Chapter 5, ethylene and sulfur monochloride give a mixture of dichloroethyl sulfide and polysulfide. With higher unsaturates the reaction is by no means so clean cut, but it may be assumed that a chlorinated sulfide is the initial product.^{6b, 391, 480, 520, 553, 577, 595, 620, 695, 803, 883, 888, 958, 1049, 1215b, 1281, 1282, 1333}

SULFUR AND RUBBER

The most important case of the action of sulfur on a hydrocarbon is the vulcanization of rubber which has been practiced industrially for over a hundred years, but which is not yet understood. A discussion even of what is known about the process would lead too far. Several references are given out of many hundreds that might be cited.^{219, 558, 559, 560} This subject will be treated again in Volume III where thioelastomers are considered. A plausible structure for ebonite, the end product of the action of sulfur on rubber, has been derived from pyrolysis experiments.⁸⁹⁷

Mono- and Divinyl Sulfides

These are of interest on account of their reactions which include polymerization.

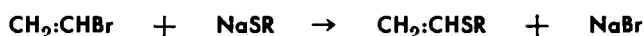
Vinyl sulfide, $(\text{CH}_2:\text{CH})_2\text{S}$, was described by Semmler¹¹⁷¹ though the boiling point, 101° , which he gave for it, was too high. It was obtained later by hydrolyzing mustard gas with alcoholic alkali.^{66a, 66b, 346, 563} It is one of a number of products from the treatment of mustard gas with zinc dust.^{735a, 735b} An improved preparation method has been given.¹¹²³

It is claimed that vinyl, allyl, cyclohexenyl, and other unsaturated sulfides can be obtained by dehydrating hydroxy sulfides.^{375b}

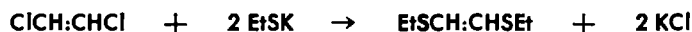
Vinyl sulfide, when first obtained, is miscible with alcohol but in a few hours it is no longer so. It polymerizes to an opaque jelly which can be converted to a friable powder by treatment with alcohol or acetone.³⁴⁶ A number of copolymers with other unsaturates have been claimed.^{386, 1374, 1392} Its copolymerization can be modified by a mercaptan.³¹⁸ Vinyl sulfide takes up one or two molecules of hydrogen chloride to form α -chloroethyl vinyl sulfide, MeCHClSCH:CH_2 , or α,α' -dichloroethyl sulfide, $(\text{MeCHCl})_2\text{S}$. Unlike its isomer, this is not vesicant. The addition of one molecule of hydrogen chloride and then one of hydrogen bromide gives MeCHClSCHBrMe .^{66a, 66b} Vinyl sulfide takes up one or two molecules of chlorine to form $\text{ClCH}_2\text{CHClSCH:CH}_2$, or $\text{ClCH}_2\text{CHClSCHClCH}_2\text{Cl}$. Removing hydrogen chloride from these, by alcoholic potash or triethyl amine, gives β -chlorovinyl-vinyl sulfide, and $\text{ClCH}_2\text{CHClSCH:CHCl}$. Hydriodic acid and thiophenol can be added to vinyl sulfide. The products are $\text{S}(\text{CH}_2\text{CH}_2\text{I})_2$ and $\text{S}(\text{CH}_2\text{CH}_2\text{SPh})_2$.¹⁰ Vinyl sulfide reacts with the amino groups of α -aminoacids, but not so readily as does mustard gas.^{74, 175} The β,β' -dichlorovinyl sulfide is obtained by the pyrolysis of the tetrachloro compound: ^{338, 703}



Vinyl alkyl sulfides can be obtained by reacting vinyl bromide with a sodium mercaptide: ⁷⁹³



A similar reaction takes place with 1,2-dichloroethylene:



The diethyl derivative boils at 170° at 13 mm. and the diphenyl melts at 61° .^{338, 457, 460, 463}

Vinyl ethoxyethyl sulfide is found along with vinyl sulfide among the products of the action of zinc dust on mustard gas in alcohol solution. It is probably formed by the addition of alcohol to vinyl sulfide:



α -Methylvinyl ethyl sulfide is produced by the pyrolysis of a mercaptole: ¹³⁰⁶



With some mercaptols this takes place spontaneously.^{249,5}

Methyl vinyl sulfide is one of the products of the pyrolysis of dimethylmercaptoethyl sulfide, $S(CH_2CH_2SMe)_2$.²⁸¹ It can be prepared by refluxing methyl β -chloroethyl sulfide in amyl alcohol in which sodium has been dissolved: ^{221, 1030}



It combines with mercaptans.^{221, 281} Butyl vinyl sulfide has been made similarly.^{1366b} Ethyl vinyl sulfide has been obtained by the pyrolysis of β -ethylmercaptocrotonic acid.^{41a} β -Phenylmercaptocrotonic acid, $MeC(SPh):CHCO_2H$, and the corresponding benzyl acid, $MeC(SCH_2Ph):CHCO_2H$, lose carbon dioxide to give phenyl α -methylvinyl sulfide, $PhSCMe:CH_2$, and benzyl α -methylvinyl sulfide, $PhCH_2SCMe:CH_2$, respectively.^{41b, 398} The reaction of ethylene oxide with mercaptans produces alkylmercaptoethanols, $RSCH_2CH_2OH$, which can be dehydrated to alkyl vinyl sulfides, $RSCH:CH_2$, which polymerize readily.^{375a} Phenyl vinyl sulfide is obtained by the pyrolysis of the diphenyl derivative of ethanedithiol: ^{205, 744a}



It is formed in 90% yield when β -phenylmercaptoethyldimethylphenylammonium iodide, $PhSCH_2CH_2NMe_2PhI$, is heated with alkali.^{194a}

Methylmercaptoethyl vinyl sulfide, $MeSCH_2CH_2SCH:CH_2$, has been obtained by distilling the sulfonium salt from dithiane and methyl iodide with alkali. The ethyl compound resulted from the chlorosulfide and alkali: ³⁵⁵

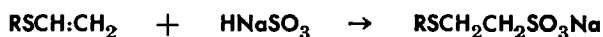


The unsaturated sulfides, $CH_2:CHCH_2CHMeSMe$ and $MeCH:-CHCH_2CH_2CH_2SMe$, are from the pyrolysis of cyclic sulfonium hydroxides.⁵¹⁷

The vinyl alkyl sulfides are of interest since the vinyl group is activated by the sulfur so that additions to them are made readily. Examples of the addition of mercaptans and of alcohols have been given already.^{629c, 632a, 637a, 1086b} Spontaneous addition with evolution of heat may take place even without a catalyst: ^{1204, 1232}



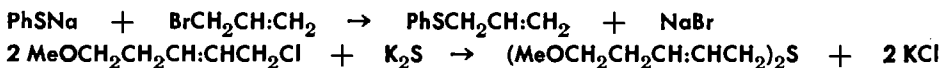
According to the nature of R and R', the products may have various uses.^{631d, 637c, 1086a} Vinyl alkyl, or aryl, sulfides, in which the second group may be ethyl, hexyl, dodecyl, cyclohexyl, phenyl, or benzyl, combine with sodium bisulfite: ^{630b}



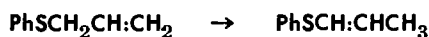
They may be polymerized alone or with other compounds.^{634a, 639, 1033, 1089} Alkylmercaptostyrenes, *p*-RSC₆H₄CH:CH₂, polymerize satisfactorily.⁴⁷

Vinyl alkyl, or aryl, sulfides can be oxidised to the sulfoxides or sulfones.^{460, 771b, 1233} These are claimed as useful intermediates.^{632c, 640, 1311}

Unsaturated sulfides of another type are made from mercaptans and halides of the allyl class: ^{53, 325, 352.5, 626, 738, 1041, 1274}

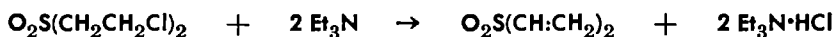


Allyl sulfide is prepared similarly.^{56, 471, 1042} The allyl phenyl sulfide is partially isomerized to *o*-allylthiophenol at 200°. ^{626, 1274} Refluxed with sodium ethylate in ethanol, allyl phenyl sulfide is isomerized: ¹²⁶⁷



The ultraviolet ¹⁰³³ and Raman spectra of several sulfides containing the vinyl group have been studied.^{352, 712, 718}

Vinyl sulfone, (CH₂:CH)₂SO₂, is one of the products obtained when zinc dust is added to a refluxing alcoholic solution of β,β'-dichlorodiethyl sulfone, (ClCH₂CH₂)₂SO₂.^{735a, 735b} To prepare it, triethyl amine is added to this sulfone in benzene: ^{10, 1212}



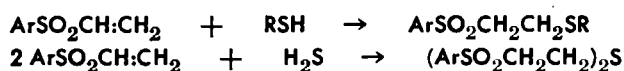
It boils at 120–1° at 18 mm. and has a density of 1.1790 at 20°. Its reactions are much like those of vinyl sulfide: Hydrobromic and hydriodic acids, water, alcohols, and phenol are taken up to form O₂S(CH₂CH₂Br)₂, m. 109°, O₂S(CH₂CH₂I)₂, m. 205°, O₂S(CH₂CH₂OH)₂, and O₂S(CH₂CH₂OR)₂.^{10, 735a, 735b} Thiophenol and mercaptoethanol unite with vinyl sulfone to make O₂S(CH₂CH₂SPh)₂ and O₂S(CH₂CH₂SCH₂CH₂OH)₂.¹²¹² Alkyl vinyl sulfones, RSO₂CH:CH₂, are made by removing hydrochloric acid from chlorides, RSO₂CH₂CH₂Cl.^{744b} The tetrabromide,

$(\text{BrCH}_2\text{CHBr})_2\text{SO}_2$, melts at 138° . Vinyl sulfone polymerizes on standing. Vinyl sulfoxide, $\text{OS}(\text{CH}:\text{CH}_2)_2$, takes up methanol in the presence of sodium methylate:



The product boils at $164-5^\circ$ at 17 mm.¹⁰

Vinyl alkyl, or aryl, sulfones take up mercaptans or hydrogen sulfide: ^{631c, 1088}



Cyclopentadiene unites with allyl benzyl sulfide²²⁶ to form a dicyclo compound.

Vinyl selenide and the acetylene derivatives, $(\text{HC}:\text{C})_2\text{Se}$, $(\text{MeC}:\text{C})_2\text{Se}$, and $(\text{EtC}:\text{C})_2\text{Se}$, have been claimed as antiknock agents in fuel.³⁷¹

CHAPTER 2.

Reactions of Sulfides

Introduction

Ethers are relatively unreactive and their reactions are comparatively few and uninteresting. Not so with sulfides; they react readily with many reagents.

Addition Compounds

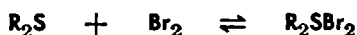
Ethyl sulfide dissolves in anhydrous hydrofluoric acid, apparently forming $\text{Et}_2\text{S}\cdot\text{HF}$, which may be regarded as a sort of sulfonium salt. Dilution with water liberates the sulfide.^{701, 879} * Liquid hydrofluoric acid extracts the lower alkyl sulfides from their solutions in hydrocarbons.⁷⁷⁷ Conversely, hydrofluoric acid may be absorbed out of a gaseous mixture by a bis-sulfide, $\text{EtSCH}_2\text{CH}_2\text{SEt}$.^{825b} Certain aliphatic sulfides combine with hydrofluoric acid and cuprous fluoride to form liquids of the composition $3\text{R}_2\text{S}\cdot 3\text{HF}\cdot\text{CuF}$.^{825c} At low temperatures, methyl sulfide absorbs hydriodic acid to form crystalline $\text{Me}_2\text{S}\cdot\text{HI}$. It reacts similarly with hydrobromic acid.^{245d, 246c, 246d} The lower mercaptans, and alkyl sulfides and disulfides are soluble in concentrated sulfuric acid, but of these the sulfides are the most soluble.⁸⁷⁹ Methyl sulfide and boron hydride form an addition compound, $\text{Me}_2\text{S}\cdot\text{BH}_3$, which melts at -38° .^{231.5} Ethyl sulfide

* As was stated at the beginning of Chapter 1, the references in this chapter have been combined with those of Chapters 1 and 3 and are found under "Bibliography" at the end of Chapter 3.

forms a compound, $\text{Et}_2\text{S}\cdot\text{SO}_2$, with sulfur dioxide.⁹ Methyl sulfoxide is an excellent solvent for sulfur dioxide.¹¹⁹⁵ Alkyl sulfides form crystalline adducts with urea.⁴²⁰

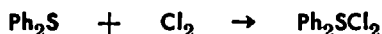
WITH HALOGENS

The primary reaction of chlorine and bromine with alkyl sulfides is addition:



With the simpler alkyl sulfides, this addition is quantitative and, as will be shown in the analytical section, may be used for their estimation. As the reaction is reversible, these compounds are excellent halogenating agents. This halogenation frequently involves the alkyl or aryl group of the sulfide.

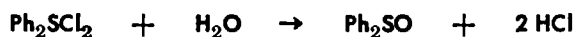
Alkyl sulfides take up halogens readily, aryl sulfides somewhat less so. Diphenyl sulfide takes up chlorine:



The chlorine migrates to the ring. The *p,p'*-dichlorodiphenyl sulfide takes up more chlorine which is given off on heating:



These chlorides are readily hydrolyzed: 449, 723b



The hydrolysis is more or less reversible. Dibenzyl sulfoxide is converted by hydrogen chloride into the hydroxy-chloride:



These chlorides act like salts of a weak base. Diphenyl sulfide is deodorized by treatment with chlorine or bromine.³⁷²

The oxidation of sulfides to sulfoxides by chlorine, in the presence of water, must involve addition of chlorine and hydrolysis.^{131b, 1102, 1413} In the determination of iodine numbers of unsaturates containing sulfide linkages, the addition of halogen to the sulfur must be taken into account. This is important in estimating the unsaturation in the vulcanization of rubber.^{130a, 956}

Just what happens in a particular case depends on the conditions of the chlorination. Diverse products may be obtained as is illustrated in the examples given. This applies also to bromination. When chlorine is passed into ethyl sulfide, much heat is

evolved; ethyl chloride is produced and sulfur separates.^{1001a} Propyl sulfide gives 1,1,1-trichloropropane and a pentachloropropane, *i*-butyl sulfide gives 2-methyl-1,1,1-trichloropropane, a dichloro- and a tetrachlorobutane, and *i*-amyl sulfide gives 2-methyl-4,4,4-trichlorobutane and other chlorinated pentanes.¹²¹⁰ From ethyl sulfide, in carbon tetrachloride, below -20° , EtSCHClMe is formed. Methyl ethyl sulfide gives MeSCHClMe and methyl benzyl sulfide yields MeSCHClPh. Methyl *i*-propyl and phenyl *i*-propyl sulfides go to MeSCMe:CHCl and PhSCMe:-CHCl which are derived from the primary products by the loss of hydrogen chloride.¹³⁹

The addition of halogens to RSC:CSR and to RSCH:CHSR is slow due to the presence of the sulfur atoms.⁴⁶³

The chlorination of certain sulfides will be discussed in Chapter 5.

The iodine of *p*-iodophenyl methyl sulfide takes up chlorine while the methyl undergoes substitution. The product is *p*-Cl₂IC₆H₄SCCl₃.¹⁴⁰⁹ The dimethyl derivative of dithioresorcinol may undergo substitution in the ring or in a methyl group.¹⁴¹²

By chlorination in dilute acetic acid solution, butyl and phenyl sulfides are converted into the sulfone chlorides, BuSO₂Cl and PhSO₂Cl.^{755b} Certain benzyl nitrophenyl sulfides are cleaved by chlorine in acetic acid solution below 30° . The 4-nitrophenyl gives the sulfone chloride, NO₂C₆H₄SO₂Cl, and the 2,4-dinitrophenyl the sulfene chloride, (NO₂)₂C₆H₃SCl.⁶³

The addition of bromine has been more thoroughly studied. The addition ^{246a, 260a, 449, 455d, 723b, 1055a} and hydrolysis follow the same pattern as with chlorine.^{428, 449, 455d, 462, 542} The hydrolysis is reversed by an excess of hydrogen bromide in a dry solvent: ^{428, 455d, 462}

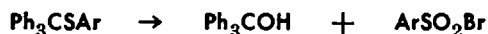


The bromine addition compound of diphenyl sulfide, Ph₂SBr₂, is only moderately stable; the bromine, like chlorine, migrates into the ring: ^{131b, 169b, 170, 373b, 729}



Methyl phenyl sulfide takes up bromine which migrates to the ring.^{170, 611, 1407b} The bis-sulfides, *p*-C₆H₄(SMe)₂, *p*-C₆H₄(SEt)₂,

and $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{Ph}$, take up four atoms of bromine each. The tetrabromide of the first exists in two forms.^{101b, 1407a} With excess bromine, substitution may take place with an alkyl sulfide.⁹⁰² In the presence of water, an aryl triphenylmethyl sulfide is cleaved and the fragments oxidised by bromine:⁵¹⁴

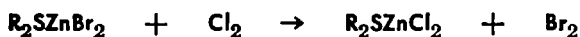


The tetrasulfides, $\text{C}(\text{SMe})_4$ and $\text{C}(\text{SPh})_4$, take up two molecules of bromine instead of four.^{55b, 55c}

A dialkyl thiobromide reacts with zinc:



The bromine in this is displaced by chlorine:^{978a, 978b}



The alkyl sulfide dibromides, R_2SBr_2 , resemble the oxonium dibromides of ethers, R_2OBr_2 , though the heats of formation of the sulfur compounds are greater than those of the oxygen compounds.²⁸⁴

Iodine also is taken up by alkyl sulfides.^{455d, 978b, 1055a, 1055b, 1407a} The dimethyl sulfide iodide, Me_2SI_2 , is like iodine in appearance. It reacts with silver cyanide:^{978b}



The double sulfide, $\text{MeSCH}_2\text{CH}_2\text{SMe}$, combines with bromine triiodide, BrI_3 , which makes four atoms of halogen to two of sulfide sulfur.⁹⁰⁹ The complexes which iodine forms with alkyl sulfides have unique absorption bands at about 308 mμ in the ultraviolet. As little as 1 part per million of an aliphatic sulfide can be detected.⁵⁵⁴ When alkyl sulfides are heated with iodine to 120° in sealed tubes, sulfonium iodides are formed, Me_3SI from methyl sulfide and Me_2EtSI from a mixture of the ethyl and methyl sulfides.^{260a, 262a} Benzyl sulfide and iodine at 120° in acetic acid form the diiodide, m. 63°, which passes into the tetraiodide.⁴¹³ A trace of iodine aids the oxidation of benzyl sulfide to the sulfoxide by air in the presence of light.^{584b} Alkyl sulfides, as well as sulfide ions, catalyze the decomposition of azide ions by iodine. This must involve the reaction of the iodine with the sulfide.⁴⁵

When an alkyl sulfide is oxidised by nitric acid, a product can

be isolated having the composition, R_2SOHNO_3 . This is probably a basic nitrate, $R_2S(OH)NO_3$. It is hydrolyzed by water:



The sulfoxide, R_2SO , is an extremely weak base and its nitrate, like its bromide, R_2SBr_2 , is readily hydrolyzed.^{462, 542, 952a, 1137c} The basic nitrate formula has been proposed for a selenium compound, $Me_2Se(OH)NO_3$.⁶⁵³

Alkyl and aryl selenides and tellurides take up halogens similarly. Dimethyl selenium chloride, Me_2SeCl_2 , melts at 59.5° and the bromide, Me_2SeBr_2 , at 82° with decomposition. The iodide, Me_2SeI_2 , has been prepared. The chloride, Et_2SeCl_2 , is unstable.^{653, 1055b} Several aryl compounds are known: $(\alpha-C_{10}H_7)_2SeBr_2$ m. 183° , $(\beta-C_{10}H_7)_2SeCl_2$ m. 146° , $(\alpha-C_{10}H_7)_2TeCl_2$ m. 265° , $(\alpha-C_{10}H_7)_2TeBr_2$ m. 244° , and Ph_2TeCl_2 m. 146° .⁸¹⁵ Diphenyl selenium chloride, Ph_2SeCl_2 , is made by the action of hydrochloric acid on the selenoxide, Ph_2SeO , which is from the hydrolysis of the bromide.⁷²⁹ Tetrachlorodivinyl selenide takes up chlorine at low temperatures to form the unstable chloride, $Cl_2Se(CCl:CHCl)_2$, m. 87° .²⁰² Certain of these tellurium compounds can be formed in a different way:¹¹¹²



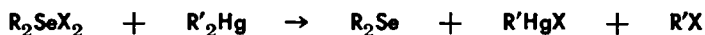
The dissociation constants of several aryl selenide dibromides have been studied:^{827, 828}



In the case of the alkyl-aryl selenide, the compound is disrupted:^{87.5}



The halogen is removed by a mercury alkyl:^{757a}



Diphenyl selenide dibromide and silver acetate give a diphenyl selenonium monoacetate, $Ph_2Se(OH)OAc$.⁴³⁸

Alkyl sulfides form compounds with chloramine-T, $R_2S:NSO_2-C_6H_4Me$, and with chloramine-B, $R_2S:NSO_2Ph$. These have distinctive melting points and are used to identify the sulfides, particularly dichloroethyl sulfide.^{152.5, 349, 778, 840, 1167, 1269} Several melting points are given:

	Me ₂ S	MeSEt	Et ₂ S	(ClCH ₂ CH ₂) ₂ S	(PhCH ₂) ₂ S
T	159° ⁷⁷⁸	133° ⁸⁴⁰	144° ⁸⁴⁰	144.5° ⁸⁴⁰	193° ^{788, 840}
B	131° ⁷⁷⁸	—	115° ⁷⁷⁸	—	153° ⁷⁷⁸

These are reaction products rather than addition compounds. The sulfur appears to be quadrivalent. Detailed directions for preparing these have been given. Hydrogenation regenerates the sulfides.⁸²⁴ One allyl group of the allyl sulfide compound migrates to the nitrogen. Compounds of this type have been prepared from other chloramines, 4,3-Me(O₂N)C₆H₃SO₂NCINa and α-C₁₀H₇SO₂NCINa.^{34, 35}

Alkyl sulfides give colored compounds with alkyl nitrites and with tetranitromethane. These appear to be intermediates which pass into stable colorless compounds.^{548, 822} Combination of an alkyl sulfide with perchloric acid is said to separate it into two forms which differ slightly in properties, but this does not seem to be generally accepted.^{583b, 584d} Methyl, ethyl, and propyl sulfides form unstable addition compounds with triphenylmethyl:¹¹⁰



Aryl sulfides can be sulfonated by the usual methods.^{1007b, 1008} Diphenyl sulfide, dissolved in sulfuric acid, is converted to the disulfonic acid on standing 4 days at room temperature.¹⁷³

ALKYL SULFIDES AND SALTS OF HEAVY METALS

One of the most characteristic properties of alkyl sulfides is their power to form addition products with salts of the heavy metals. Of these complexes, those containing platinum and mercury salts are the most numerous. When an alkyl sulfide is prepared, it is almost as natural to describe its combinations with salts of these metals as it is to give the melting point of the phenylurethane of a new alcohol. Compounds with salts of copper, silver, gold, mercury, nickel, lead, platinum, zinc, cadmium, aluminum, bismuth, titanium, and tin are reviewed in Pfeiffer's book.⁹⁹⁵ β-Naphthyl sulfide gives spot tests with silver, mercury, iron, and copper salts.^{6,5}

When allyl sulfide was isolated from oil of garlic, its complexes with salts of mercury, platinum, palladium, and silver were prepared.¹³⁵⁷ These were used to show the identity of the natural sulfide with the synthetic sulfide.⁵⁹⁶

With Mercury Salts

The mercuric chloride compounds have been particularly useful for isolating alkyl sulfides from mixtures containing them.¹¹⁷¹ An example of this was Mabery's isolation of thiophanes from high-sulfur petroleum distillates. These were treated with an alcoholic solution of mercuric chloride. The crystals were filtered off, washed, suspended in water, and decomposed by hydrogen sulfide. The recovered alkyl sulfides, which turned out to be cyclic, were identified by physical properties and analyses.^{816a, 816b, 817b} Cyclic sulfides have been isolated from a gasoline by this method.¹⁰¹⁴ Directions have been given for the identification and estimation of alkyl sulfides in petroleum.^{70, 218} The lower alkyl sulfides are more easily removed than the higher.¹⁵⁸ Mercurous acetate has been used in such separations.^{117, 5} Fractional extraction of petroleum distillates with an aqueous solution of mercurous acetate takes out cyclic sulfides preferentially.^{118a}

The usual method of preparation of the mercuric chloride-alkyl sulfide complexes is the addition of an alcoholic solution of the mercuric halide to a like solution of the alkyl sulfide. The complexes separate out and are filtered off. They usually have satisfactory melting points though their usefulness for identification is lessened by the fact that the same alkyl sulfide may form several different complexes with the same mercuric salt. The molecular ratios may be 2:1, 1:1, 1:2, or some other. In any given case, it is probable that several different complexes are in equilibrium with one another and with their components. The one that comes out is likely to be the one that is the least soluble under the particular conditions. One of them may pass into another. Thus recrystallization from ethanol changes $\text{Et}_2\text{S} \cdot \text{HgCl}_2$ into $\text{Et}_2\text{S} \cdot 2\text{HgCl}_2$.¹³⁴⁸ Propyl sulfide and mercuric chloride in alcohol give the compound $\text{Pr}_2\text{S} \cdot \text{HgCl}_2$ which is converted to $\text{Pr}_2\text{S} \cdot 2\text{HgCl}_2$ by crystallization from benzene. Methyl sulfide gives a compound $2\text{Me}_2\text{S} \cdot 3\text{HgCl}_2$ which may be a mixture.^{404b, 998b} This was formerly supposed to be $\text{Me}_2\text{S} \cdot \text{HgCl}_2$.^{796a, 796b} The mercuric chloride compound from octyl sulfide is $(\text{C}_8\text{H}_{17})_2\text{S} \cdot \text{HgCl}_2$.⁹⁰² The addition compound with thioxane is $\text{O}(\text{CH}_2\text{CH}_2)_2\text{S} \cdot \text{HgCl}_2$ m. 171° .³⁰⁶ Aromatic sulfides and mercuric chloride combine in the same ratio regardless of the solvent or of the proportions of the components: $(\text{PhCH}_2)_2\text{S} \cdot \text{HgCl}_2$, m. 136° , $(\text{PhCH}_2\text{CH}_2)_2\text{S} \cdot$

2HgCl_2 , m. 101° , $(\text{PhCH}_2\text{CH}_2\text{CH}_2)_2\text{S}\cdot 2\text{HgCl}_2$, m. 120° , and $(\text{PhCHMe})_2\text{S}\cdot \text{HgCl}_2$, m. 112° .^{105b, 106} The melting points of a few of these compounds are in Table 1.2:

TABLE 1.2
*Melting Points of Some Alkyl Sulfide-Mercuric
Chloride Addition Compounds*^{404b}

Sulfide	1 HgCl_2	2 HgCl_2
Et_2S	77° , 90° ⁶⁶⁸	119.5°
Pr_2S	88°	122° , 127.5° ⁸³¹
<i>i</i> - Bu_2S	116°	131°
<i>n</i> - Bu_2S	—	113° , 110.5° ⁸³¹
MeEtS	—	128° ²⁷⁷
MeBuS	—	116.5° ¹²²
MeAmS	—	127° ¹²²
<i>i</i> - BuEtS	—	108° ⁶⁴⁷
<i>i</i> - AmEtS	—	87° ⁶⁴⁷
EtSCH_2Ph	84° ¹¹³⁰	142° ¹¹³⁰

Multiple sulfides, such as $\text{EtSCH}_2\text{CH}_2\text{SCH}:\text{CH}_2$ ³⁵⁵ and $\text{MeSCH}_2\text{CH}_2\text{SMe}$,^{909, 910} form complexes with mercuric chloride. The latter seems to have this power to an unusual degree, which is attributed to chelation with the two sulfur atoms. Only one molecule of mercuric chloride is taken up.⁹⁰⁹ The sulfides, $\text{C}(\text{CH}_2\text{SR})_4$, combine with two molecules of mercuric chloride, one for each pair of sulfur atoms. The melting points of $\text{C}(\text{CH}_2\text{SR})_4\cdot 2\text{HgCl}_2$ are Me 143° , Et 157.5° , Pr 88° , Bu 97° , and Am 84° .^{51c} The tetramethyl compound, $\text{C}(\text{SMe})_4$, combines with 4HgCl_2 .^{55b}

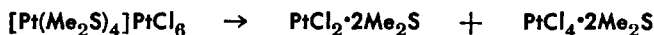
Mercuric iodide also forms complexes with alkyl sulfides.^{785, 1197a, 1352, 1366b} Finely powdered mercuric sulfide, alcohol, and ethyl iodide, heated together, give the compound $\text{Et}_2\text{S}\cdot \text{HgI}_2$, m. 110° .^{796a, 796b}

Double compounds of alkyl tellurides and mercuric salts, $\text{R}_2\text{Te}\cdot \text{HgX}_2$, are formed quantitatively from the components. $\text{Me}_2\text{Te}\cdot \text{HgCl}_2$ melts at 179° , the bromide at 161° , and the iodide at 107° . These decompose in moist air, liberating the alkyl telluride.²⁵⁹

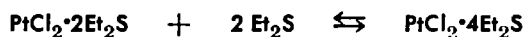
With Platinum Salts

The numerous and varied complexes which alkyl sulfides form with platinum salts have been studied extensively.^{4, 19, 127b, 128, 129, 168, 289, 290, 291, 292, 293, 295, 296, 297, 298, 379, 380, 450, 451, 600, 613, 655, 700c, 700e, 796a, 839, 1060b, 1065a, 1066, 1067, 1069, 1071a, 1171, 1209, 1236b, 1350, 1352, 1357} A common type of complex is $\text{PtX}_2 \cdot 2\text{R}_2\text{S}$ ^{292, 297, 600, 613} which can be halogenated to $\text{PtX}_4 \cdot 2\text{R}_2\text{S}$. The melting points of a number of these are in Table 2.2.

There are said to be four varieties of the compound $\text{PtCl}_2 \cdot 2\text{Et}_2\text{S}$, melting at 96° , 104° , 108° , and 110° .¹⁰⁶⁹ It is claimed that the valence of platinum varies in the series, $\text{PtCl} \cdot \text{Et}_2\text{S}$, $\text{PtCl}_2 \cdot 2\text{Et}_2\text{S}$, $\text{PtCl}_3 \cdot 2\text{Et}_2\text{S}$, $\text{PtCl}_4 \cdot 2\text{Et}_2\text{S}$, and $\text{PtCl}_5 \cdot 2\text{Et}_2\text{S} \cdot \text{H}_2\text{O}$.^{1065a, 1069} One complex may split into two others:²⁸⁹



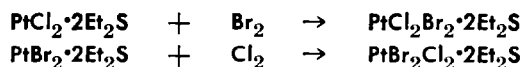
There is said to be an equilibrium:²⁹⁸



There are two forms each of $\text{PtCl}_2 \cdot \text{Et}_2\text{S}$, $\text{PtBr}_2 \cdot \text{Et}_2\text{S}$, and $\text{PtI}_2 \cdot \text{Et}_2\text{S}$.^{19, 700c, 700e} This has been explained by assuming that the four valences of platinum are in a plane.^{19, 380} The resolution of β -aminoethyl sulfide platinum chloride complex⁸³⁹ is important since it appears to show that the coordinate bond is as real as an electrocovalent or a covalent bond. The stereoisomerism of the platinum-alkyl sulfide complexes is far too complicated to be discussed here in any detail. Working out the configurations and accounting for all of the isomers is a difficult task.^{19, 295, 380, 655, 700c, 700e, 1067, 1350}

The interfacial tension between water and 1% benzene solutions of the *trans* and *cis* isomers of $\text{PtCl}_2 \cdot 2\text{Et}_2\text{S}$ are 35.30 and 28.62, respectively.⁴ The dipole moments of a number of pairs of the $\text{PtX}_2 \cdot 2\text{R}_2\text{S}$ compounds have been determined.⁶⁵⁵

The dihalides are readily converted to the tetrahalides:



The same product is obtained in the two reactions. This has been interpreted as indicating a tetrahedral structure for the alpha compounds.^{19, 380}

TABLE 2.2

Melting Points of Complexes: PtX₂·2R₂S and PtX₄·2R₂S

Sulfide	PtCl ₂	PtBr ₂	PtI ₂	PtCl ₄	PtBr ₄	PtI ₄
2 Me ₂ S	α 159° 127b, 1350	159° 1350	—	—	—	—
2 Et ₂ S	β 106° 127b, 1350	118° 127b	136° 127b	175° 127b	—	104° 127b
2 Pr ₂ S	a	105° 129, 1350	133° 129, 1350	139° 129	141° 129	—
2 Bu ₂ S	b	65° 128	67° 128	—	—	—
2 i-Bu ₂ S	c	144° 128	187° 128, 1350	162° 128	184° 128	—
(·CH ₂ SMe) ₂	226° 295	—	—	—	—	—
(·CH ₂ SEt) ₂	188° 295	—	—	—	—	—
(·CH ₂ SPr) ₂	255° 295	—	—	—	—	—
(CH ₂) ₃ (SEt) ₂	135° 295	—	—	—	—	—

a α-m.46°, β-m.86° 1850, b α-m.40°, β-m.77° 128, c α-m.83° 128 β-m.139° 129, 1350. PtBr₂Cl₂·2Pr₂S m.129°; PtCl₂Br₂·2Pr₂S m.129° 129.

Allyl sulfide and platinum sulfide unite in several different proportions to form complexes.¹³⁵⁷

Methyl selenide forms a complex, $(\text{Me}_2\text{Se})_2\text{PtCl}_4$, with platinic chloride.⁶⁵³ Other alkyl selenides do the same.^{452a} The conductivities of the platinum and palladium complexes have been studied.^{452b}

With Palladium Salts

Considerable attention has been given to complexes of palladium salts with organic sulfides.^{24, 293, 294, 379, 450, 647, 648, 841, 910, 998a, 1357} One molecule of palladium chloride, bromide, iodide, nitrate, sulfate, or oxalate combines with two of an alkyl sulfide. The melting points of a number of the compounds are in Table 3.2.

TABLE 3.2

Melting Points of Palladium Halide-Alkyl Sulfide Complexes

Sulfide	PdCl_2	PdBr_2	PdI_2	$\text{Pd}(\text{NO}_3)_2$	$\text{Pd}(\text{NO}_2)_2$
$2\text{Me}_2\text{S}$	130° ²⁴	125° ²⁴	—	138° ⁸⁴¹	155° ²⁴
$2\text{Et}_2\text{S}$	83° ³⁷⁹	100° ²⁹⁴	104° ²⁹⁴	163° ⁸⁴¹	145° ²⁴
$2\text{Pr}_2\text{S}$	59° ⁸⁴¹	—	—	164° ⁸⁴¹	—
$2\text{Bu}_2\text{S}$	32° ⁸⁴¹	—	—	166° ⁸⁴¹	—
$2\text{-}i\text{-Am}_2\text{S}$	95° ²⁴	133° ²⁴	143° ²⁴	—	180° ²⁴
$(\cdot\text{CH}_2\text{SMe})_2$	235° ⁹¹⁰	220° ⁹¹⁰	204° ⁹¹⁰	—	—
$(\cdot\text{CH}_2\text{SEt})_2$	182° ³⁷⁹	159° ²⁹⁴	154° ²⁹⁴	—	—

Other compounds have been prepared: $\text{PdCl}_2 \cdot 2i\text{-Bu}_2\text{S}$, m. 163°, $\text{PdCl}_2 \cdot 2\text{Am}_2\text{S}$, m. 41°, the heptyl and octyl are oils, $\text{PdCl}_2 \cdot 2\text{MeEtS}$, m. 67°, $\text{Pd}(\text{NO}_3)_2 \cdot 2\text{Am}_2\text{S}$, m. 163°.⁸⁴¹ The mixed compounds, $\text{PdCl}_2 \cdot \text{Am}_2\text{S} \cdot \text{Bu}_2\text{S}$ and $\text{PdCl}_2 \cdot \text{Et}_2\text{S} \cdot \text{Am}_2\text{S}$, have been prepared.²⁴ $(i\text{-BuSBu})_2 \cdot \text{PdCl}_2$, m. 73.5° and $[(i\text{-PrMeCH})_2\text{S}]_2 \cdot \text{PdCl}_2$ have been reported.⁶⁴⁷ Phenyl alkyl sulfides, PhSR , also combine readily with palladium chloride and give good yields of the complexes, $\text{PdCl}_2 \cdot 2\text{PhSR}$. A number of these have been prepared. R = Et m. 140°, Pr 91°, *i*-Pr 162°, Bu 106.5°, *i*-Bu 93.5°, *s*-Bu 138°, Am 76°, *i*-Am 97°, act. Am. oil, *t*-Bu 84°, *t*-Am 73°.^{647, 648}

When heated, the compound, $\text{PdCl}_2 \cdot 2\text{Me}_2\text{S}$, decomposes into palladium sulfide and methyl sulfide and chloride. It is reduced by hydrogen to methyl chloride, hydrogen chloride, and palladium.^{998a, 998b}

With Copper Salts

Cuprous chloride forms an unstable complex with methyl sulfide.^{998a, 998b, 1352} Benzyl sulfide combines with cuprous chloride, bromide, and iodide, $\text{CuCl} \cdot 2(\text{PhCH}_2)_2\text{S}$, m. 98.3° , the bromide melts at 87.8° , and the iodide at 63.5° .⁶¹³ The dimethyl derivative of ethane dithiol, $\text{MeSCH}_2\text{CH}_2\text{SMe}$, combines with cuprous and cupric salts, and also with salts of a number of other metals. The possibility of the formation of a chelate five-membered ring appears to favor the formation of these.^{288a, 910} The heats of combination of a molecule of the dimethyl derivative of ethane dithiol with a molecule of the salt are given in Table 4.2 in kilogram calories.⁹⁰⁹ Cupric chloride is a selective adsorbent

TABLE 4.2

Heats of Combination of $\text{MeSCH}_2\text{CH}_2\text{SMe}$ with Salts

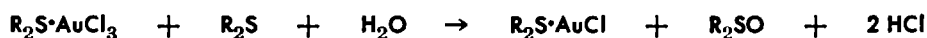
CuCl_2	14.85	CdCl_2	12.16	HgI_2	4.83
CuI	5.88	CdBr_2	11.27	SnCl_4	28.06
ZnCl_2	13.19	CdI_2	9.96	SnBr_4	19.40
ZnBr_2	15.35	HgCl_2	13.23	SnI_2	16.58
ZnI_2	14.18	HgBr_2	11.33		

for dialkyl monosulfides in anhydrous hydrocarbon solution.¹¹⁵⁹ Ethyl and phenyl sulfides in illuminating gas cause corrosion of copper needle valves.⁹²³ Complexes of alkyl sulfides with copper salts of organic acids have been patented.^{949c, 991}

With Gold Salts

A number of complexes of alkyl sulfides with gold chlorides have been prepared: $\text{AuCl}_3 \cdot \text{R}_2\text{S}$, in which R may be methyl, ethyl, propyl, *i*-butyl, and benzyl, and $\text{AuCl} \cdot \text{R}_2\text{S}$.^{573, 998a, 998b, 1073, 1199} Benzyl sulfide combines with several gold halides.⁶¹³ The reported $(\text{PhCH}_2)_2\text{S} \cdot \text{AuBr}_2$ appears to be a mixture of aurous and auric compounds.¹⁷⁹ The cyclic dithiane combines with gold chlorides in several proportions.^{1060a, 1065b} The sulfide bromide, Me_2SBr_2 , gives compounds from which bromine may be washed out.¹²⁰⁹ The double sulfide, $\text{MeSCH}_2\text{CH}_2\text{SMe}$, combines with aurous chloride and also with auric chloride.^{293, 910}

The reaction of an alkyl sulfide with auric chloride takes place in two steps. The first is the formation of the colored complex, $R_2S \cdot AuCl_3$, and the second is the dechlorination of this to the colorless $R_2S \cdot AuCl$ by the alkyl sulfide. The chloride, R_2SCl_2 , which may be considered as an intermediate, is hydrolyzed to the sulfoxide: ^{573, 975, 1097}



The sulfoxide has been isolated in the case of benzyl sulfide. Alkyl sulfoxides give voluminous white precipitates with phosphotungstic acid.⁹⁷⁵ This probably accounts for the precipitates formed when a sulfide is added to auric chloride and phosphotungstic acid.^{975, 1097}

With Silver Salts

Allyl sulfide combines with two molecules of silver nitrate,^{804, 1171} while other alkyl sulfides take only one. A number of these complexes have been prepared: $AgNO_3 \cdot Me_2S$, m. 126°, $AgNO_3 \cdot Et_2S$, m. 122°, $AgNO_3 \cdot Pr_2S$, m. 109°, $AgNO_3 \cdot Bu_2S$, m. 98, and $AgNO_3 \cdot (PhCH_2)_2S$, m. 93–5°. ¹⁰⁶³ $AgNO_3 \cdot 2(PhCH_2)_2S$ melts at 105.5°. ⁶¹³

With Salts of Other Metals

Stannic chloride combines with two molecules of an alkyl sulfide¹³⁵³ or with one of a double sulfide,^{909, 910} so does stannic bromide: $SnCl_4 \cdot 2Me_2S$, $SnCl_4 \cdot 2Et_2S$, m. 102°, $SnCl_4 \cdot 2i-Am_2S$, m. 64°, $SnBr_4 \cdot 2Me_2S$, m. 87°, $SnBr_4 \cdot 2Et_2S$, m. 84°, $SnBr_4 \cdot 2i-Am_2S$, m. 46°, ¹³⁵³ $SnBr_4 \cdot MeSCH_2CH_2SMe$, m. 199°, $SnI_4 \cdot MeSCH_2CH_2SMe$, m. 100°, and $SnCl_4 \cdot MeSCH_2CH_2SMe$, m. 209°. ⁹¹⁰

o-Methylmercaptotriphenyl carbinol gives complexes with stannic and ferric chlorides.^{185b} Benzyl sulfide,⁴³⁷ ethyl sulfide, and dithiane^{1071b} give complexes with ferric chloride.

Dicarbonyl compounds of ferrous iodide and bromide with diethyl-dithioglycol ether have been described: $Fe(CO)_2I_2 \cdot EtSCH_2CH_2SEt$ and $Fe(CO)_2Br_2 \cdot EtSCH_2CH_2SEt$.⁵⁷⁸ Alkyl sulfides, methyl to amyl, give a color reaction with sodium nitroprusside; phenyl and benzyl sulfides do not.^{105a}

Cadmium iodide unites with methyl sulfide¹³⁵² and with bis-(methylmercapto)ethane.⁹¹⁰ Cadmium iodide, bromide, and chloride form complexes with dithiane.^{168, 909} Antimony chloride

forms complexes with methyl, ethyl, propyl, and butyl sulfides.^{1084b}

Several irridium chloride complexes have been prepared: $\text{IrCl}_3 \cdot 3\text{Me}_2\text{S}$, m. 239° , $\text{Ir}_2\text{Cl}_5 \cdot 4\text{Me}_2\text{S}$, $\text{IrCl}_3 \cdot 2\text{Me}_2\text{S}$, $\text{IrCl}_3 \cdot 3\text{Et}_2\text{S}$, m. 131° , $\text{Ir}_2\text{Cl}_5 \cdot 4\text{Et}_2\text{S}$, m. 207° , and $2\text{IrCl}_2 \cdot 3\text{Et}_2\text{S}_2$.^{451, 1061b} There are also rhodium complexes.⁴⁵¹ Ethyl sulfide forms them with rhodium chloride, m. 126° , with rhodium bromide, m. 107° , and with rhodium iodide, m. 98° .³⁸⁵

Bis(methylmercapto)ethane gives the complex, $3\text{MeSCH}_2\text{CH}_2\text{SMe} \cdot 2\text{BiI}_3$.⁹¹⁰ The ethyl compound, $\text{EtSCH}_2\text{CH}_2\text{SEt}$, forms complexes with nickel thiocyanate^{288a} and with bismuth salts.²⁹³

The double sulfide, $\text{MeSCH}_2\text{CH}_2\text{SMe}$, combines with zinc chloride and iodide: $\text{C}_4\text{H}_{10}\text{S}_2 \cdot \text{ZnCl}_2$, m. 132° and $\text{C}_4\text{H}_{10}\text{S}_2 \cdot \text{ZnI}_2$, m. 170° .⁹⁰⁹

Alkyl sulfides give addition compounds with titanium tetrachloride.³⁵⁴ Phenyl and alkyl sulfides form complexes with aluminum chloride.^{131a, 345, 775a} *i*- $\text{Bu}_2\text{S} \cdot \text{BCl}_3$ has been reported also.⁹⁹⁹

Decomposition of Sulfides

This usually involves the breaking of carbon-to-sulfur bonds which has been reviewed by Tarbell and Harnish.^{1266a}

According to Kékulé, thiophene results when ethyl sulfide is passed through a hot tube, but a repetition of this experiment gave low yields.^{893a} The thermal decomposition of ethyl sulfide has been studied over the range 380 to 410° .^{780, 1302} It begins to decompose at 400° .¹¹²⁰ It is difficult to remove from gases even with catalysts.⁴⁶⁸ In catalytic cracking, alkyl sulfides are destroyed while thiophenes are untouched at 475° .⁵³⁵ The cracking of butyl sulfide in naphtha is slow below 450° . The amount of butyl mercaptan formed is greater if hydrogen sulfide is added.⁸³⁵ At 230° in kerosene in 2 hours, in the presence of molybdenum sulfide, the decomposition of benzyl sulfide was 95%, of allyl sulfide, 61%, of propyl sulfide, 26%, and of *i*-amyl sulfide, 27%.⁹⁰⁴ At 496° the decomposition of *i*-amyl sulfide is much nearer to completion. A small amount of the mercaptan, thiophenes, paraffins, olefins, and hydrogen sulfide are formed.^{404a}

Nonyl and decyl sulfides give large amounts of nonene-1 and decene-1.¹²⁹⁷ Cyclopentyl sulfide goes to cyclopentene and hydrogen sulfide.¹²⁹⁵ At 260° benzyl sulfide gives stilbene and hydrogen sulfide.^{75, 456} Diphenyl sulfide gives dibenzothiophene,

benzene, and hydrogen sulfide.⁵⁰⁷ Benzhydryl sulfide decomposes at 290°: ¹¹⁵³



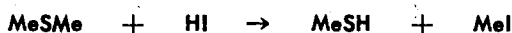
Triphenylmethyl phenyl sulfides appear to split into free radicals above 200°. ^{753a} Appearance potentials have been determined for the gaseous ions formed by electron impact on methyl and ethyl sulfides. ⁴⁴³

Over a suitable catalyst, *t*-butyl sulfide is converted by hydrogen sulfide into two molecules of the mercaptan. ^{13b} Possibly the sulfide splits into the mercaptan and isobutene which then combines with the hydrogen sulfide.

Methyl sulfide absorbs hydrogen iodide, at a low temperature, to form crystalline Me₂S·HI. When this is heated, the mercaptan and sulfonium iodide are produced:



This amounts to a cleavage of the sulfide:



Hydrobromic acid reacts similarly. ^{245c, 246c, 246d} Ethyl sulfide is split by acetyl iodide ⁵²¹ and bromide, ^{245c, 246c, 246d} but not by acetyl chloride. ⁵²¹ In general, the methylmercapto group, -SMe, is more difficult to split off from an aromatic compound than the methoxyl group, -OMe. ^{29, 1245} In the Zeisel method, with hydriodic acid, the evolution of hydrogen sulfide, along with the methyl iodide, is a complication. ¹⁰¹² When RO- and RS- groups are both present, the RO group is the one that is split off by boiling with hydrobromic acid: ¹²⁴⁴

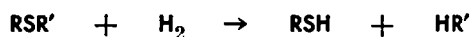


4-Hydroxyphenyl sulfide is split by hydrobromic acid into 4-bromophenol and phenyl disulfide. ⁵⁷⁹ Phenyl benzyl sulfide is cleaved by hydrobromic acid in acetic acid, or by aluminum bromide ^{547a, 1266b} or chloride, ^{755a} in benzene. The sulfur goes with the phenyl group. ¹²⁶⁸ The following phenyl aryl sulfides are arranged in the order of the ease of cleavage: Ph₃CSPH > Ph₂CHSPH > PhCH₂-SPH > PhCH₂CH₂SPH. ^{1266b} Drastic changes may be effected by aluminum chloride. ^{44a, 1305} Triphenylmethyl phenyl sulfide is split rapidly by cold concentrated sulfuric acid. ⁶⁵²

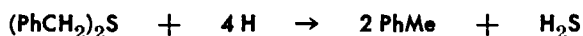
In the silent electric discharge, methyl sulfide is converted into a mixture of high-boiling liquids.⁸⁰⁰

Certain sulfide linkages can be broken even at room temperature in the presence of silver or mercuric salts at suitable *pH* values.⁹⁹³ In general, the sulfur of a monosulfide is difficult to remove, but treatment of 2,4-dinitrophenyl sulfide with dilute sulfuric acid and a metal gives *m*-phenylenediamine.⁵⁸⁷

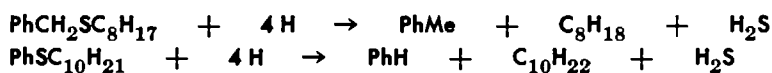
In the presence of a sulfactive catalyst, at 250° under 1500 lb./sq. in. of hydrogen, an alkyl sulfide is split into a mercaptan and a hydrocarbon: ^{408, 1190c}



Over Raney nickel, the sulfur is removed completely: ^{917, 1203}

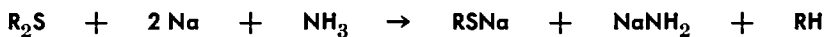


This may be accomplished with a cobalt-molybdenum catalyst: ⁶⁰⁵

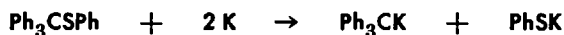


Heated in an indifferent solvent with Raney nickel, freed from hydrogen, all mercaptals of benzaldehyde, $\text{PhCH}(\text{SR})_2$, are desulfurized to stilbene. Dibenzyl is obtained from $\text{PhCH}(\text{SCH}_2\text{Ph})_2$, diphenyl sulfide from $\text{PhCH}(\text{SPh})_2$, and some diphenyl from diphenyl sulfide.⁵⁵⁶ Other sulfides are desulfurized by Raney nickel. The extent of accompanying hydrogenation depends on the hydrogen content of the nickel.^{516, 556.5, 1207} The benzyl radical is distinguished for the ease with which it can be split off by hydrogenolysis.⁹²⁰

In dry ether an alkyl sulfide is hardly attacked by sodium,⁹¹³ but in liquid ammonia it is cleaved: ^{732, 1372}



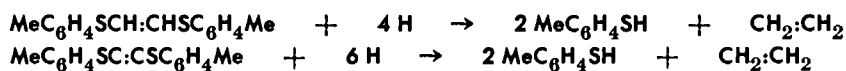
This goes particularly well with a benzyl sulfide, so well that it is used as a preparation method for mercaptans.¹¹⁸² Heated 36 hours with sodium, benzyl phenyl sulfide is broken up. Phenyl mercaptan is the chief product isolated, along with some toluene.¹¹⁸² Triphenylmethyl phenyl sulfide is cleaved by sodium or potassium: ¹⁴⁰¹



Methyl phenyl sulfide, refluxed 5 hours in dry pyridine with sodium, goes to PhSNa .^{619b}

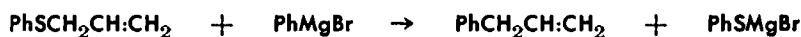
Potassium amide, KNH_2 , in liquid ammonia, may cause isomerization. Thus methyl benzyl sulfide, PhCH_2SMe , is changed to *o*-methylbenzyl mercaptan, $\text{MeC}_6\text{H}_4\text{CH}_2\text{SH}$.⁵⁵⁷

Certain sulfides are cleaved by reduction with sodium and alcohol:⁴⁶³



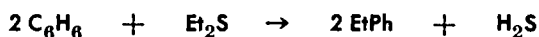
Nitrosulfides may be converted to azo-¹⁸² or azoxy-compounds¹⁸⁸ by sodium in methanol.

Diphenyl sulfoxide is decomposed by phenyl sodium. One of the products is diphenyl disulfide.⁴⁶⁵ Alkyl sulfides are stable toward Grignard reagents⁴⁹³ and can be used as solvents in their preparation.^{1215a} Phenyl allyl sulfide and phenyl magnesium bromide react when heated at 78° :



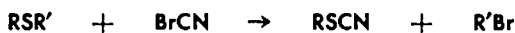
This is not a general reaction and may be attributed to the mobility of the allyl group.⁸⁰⁷

An aromatic hydrocarbon may be alkylated by passing it and a sulfide over copper phosphate at an elevated temperature:¹²⁷⁷



Benzene may be alkylated by *t*-butyl sulfide to *tertiary*-butylbenzene over a silica-alumina catalyst.⁴¹⁰

An alkyl sulfide and cyanogen bromide give a thiocyanate and an alkyl halide:

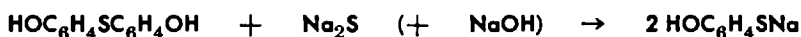


The lighter group appears in the halide unless one of the groups is benzyl in which case benzyl bromide is formed.^{193, 195, 196, 245d, 246c, 246d}

Ethyl sulfide is broken up completely by an excess of sulfur monochloride.¹⁴⁷ The sulfur linkage in aromatic sulfides is broken by sulfuryl chloride.¹⁰⁷⁶

Ethyl sulfide, in alcohol solution, does not take up any appreciable amount of sulfur, even on long standing.^{602b} Heated with sulfur at 180° , it takes up sulfur forming disulfide and polysulfide.¹⁴⁷ At 260° , with selenium as a carrier, diphenyl sulfide

and sulfur go to the disulfide and thianthrene.⁷²⁹ Ethyl sulfide and sulfur over alumina, at a high temperature, give thiophene.¹⁷⁸ Benzyl sulfide and sulfur, heated to 200°, give a mixture of 2-phenylbenzothiophene and tetraphenylthiophene.⁶¹⁰ The action of sulfur on halogenated phenyl sulfides is peculiar; 4,4'-dibromophenyl sulfide is turned into *p*-dibromobenzene.¹¹⁵ Phenyl sulfide and aqueous sodium sulfide, in an autoclave at 300°, give sodium thiophenate.^{1332b} The sulfide linkage which is usually so stable may be labilized by the presence of certain groups in particular positions. 4,4'-Dihydroxydiphenyl sulfide is split by heating with a mixture of sodium sulfide and hydroxide:^{642, 971}



p-Nitrophenyl sulfide, $(\text{NO}_2\text{C}_6\text{H}_4)_2\text{S}$, refluxed in alcohol with sodium disulfide, is changed to the disulfide, $(\text{NO}_2\text{C}_6\text{H}_4)_2\text{S}_2$.⁴⁷⁰

A method of decomposing sulfides, which has wide application, is treatment with methyl iodide. This involves the formation and decomposition of a sulfonium salt:



Any alkyl halide can be used, but methyl iodide is the most reactive.^{55a, 1150, 1170a} This reaction is mentioned again under cyclic sulfides and will be considered more fully in the section on sulfonium compounds.

Oxidation of Alkyl Sulfides

The indirect oxidation by bromine and chlorine has been discussed. The oxidisability of alkyl sulfides was one of their first properties to be observed. This goes in two stages:



Nitric acid, the most commonly used agent, effects the first stage and usually stops there.^{87, 101a, 127a, 178.5, 253, 302, 403, 457, 475, 506, 580a, 587, 622, 689, 700a, 832a, 952a, 952d, 976b, 1137a, 1137c, 1138, 1139, 1376a} Fuming nitric acid may carry the oxidation as far as the sulfone.^{302, 902, 952a, 952d, 1102, 1137a, 1137c, 1138} Electrolysis in nitric acid has been proposed.^{475, 1004} Oxides of nitrogen may be used.^{606.5, 1043b} Methyl sulfide vapor is oxidised to the sulfoxide by oxygen in the presence of oxides of nitrogen.¹¹⁹⁶

Electrolysis of benzyl sulfide in sulfuric acid gives tribenzyl-sulfonium sulfate.⁴²²

Potassium permanganate oxidises either the sulfide or the sulfoxide to the sulfone.^{42, 48, 55a, 87, 152.7, 280, 340, 434, 455a, 457, 512, 580a, 748, 866, 927, 1376a} Chromium trioxide in acetic acid^{101a, 127a, 178.5, 457, 472, 622, 707, 723c, 724, 1240} and chromate-sulfuric acid mixture are excellent for oxidising either the sulfide^{1366b, 1395} or the sulfoxide¹²⁴¹ to the sulfone. Chromic acid in acetic acid may stop at the sulfoxide.⁷⁰⁷ In some cases, only tarry products are obtained.¹²²⁹

Hypochlorite takes a sulfide all the way to the sulfone.^{1311, 1383} A sulfide may be estimated by titrating it with standard sodium hypochlorite solution with methyl orange as indicator.⁷⁵⁹ Alkaline calcium hypochlorite may destroy the sulfide, oxidising the sulfur to the sulfate ion.²³

Sulfides and disulfides can be titrated with bromide-bromate solution. The end products are, respectively, the sulfoxide, R_2SO , and the sulfone bromide, RSO_2Br .¹¹⁸⁹

Ozone is an efficient oxidising agent.^{136, 1381}

Recently the favorite oxidising agent has been 30% hydrogen peroxide. This has been used in acetone, but glacial acetic acid is preferred. When the sulfoxide is desired, one molecule of the peroxide is used to one of the sulfide; to get the sulfone, twice as much plus a slight excess is used. The sulfide and peroxide are dissolved in acetic acid and the mixture let stand overnight, after which it may be warmed.^{48, 51b, 51c, 53, 56, 105c, 161a, 178.5, 233b, 272, 279, 302, 407, 411, 416, 426, 471, 479, 484, 514.5, 580b, 583a, 583b, 622, 632c, 640, 648, 656, 664, 697, 1031.3, 1043a, 1043b, 1096, 1119c, 1202, 1233, 1311, 1402, 1406} A kinetic study of this oxidation at 30, 40, and 50° has been made.⁹⁶³ The rate of oxidation has been considered to be a measure of the chemical activity of the sulfur atom.⁶⁵⁰ The heats of oxidation to the sulfoxide and to the sulfone have been measured for methyl sulfide,³⁷⁴ the sulfone was prepared by oxidation with permanganate.

Peracetic acid has been used.⁸⁹⁶ The oxidation of phenyl sulfide to the sulfoxide by this reagent is several hundred times as fast as the subsequent oxidation to the sulfone.¹³² Oxidation may be effected by benzoyl peroxide,⁶⁰⁷ by benzoylhydroperoxide,^{771a, 771b, 772} or by phthalic monoperoxide.^{135b} Cumene hydroperoxide gives particularly good results. The reaction may take place spontaneously with evolution of heat on mixing the reactants¹⁰⁷⁹ or

it may be effected in solvents.^{722.5} The yields are practically quantitative. Either the sulfoxide or the sulfone may be obtained with selenium dioxide.⁸⁸⁴

An alkyl sulfide, notably dichloroethyl sulfide, is oxidised when exposed to oxygen in admixture with an unsaturated oil. Oxygen appears to be taken up as a peroxide, or hydroperoxide, and passed on to the sulfur.¹¹⁰⁸

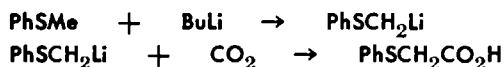
Ethyl sulfide is oxidised to the sulfone at a platinum-gauze anode.^{421, 423}

In the oxidation of sulfides, there is always a tendency to go beyond the sulfoxide and sulfone to a sulfonic acid or even to sulfuric acid. The amount of such destruction depends on the structure of the organic sulfide, on the nature of the oxidising agents, and on the conditions, such as concentration and temperature, under which the reaction is carried out. Nitric acid, though it seldom produces a sulfone, seems to favor the formation of some of the sulfonic acid.^{232, 307, 586, 700a, 1137a, 1376a, 1376b} Triphenylmethyl phenyl sulfide is oxidised by chromate to triphenylcarbinol and benzenesulfonic acid.⁵¹⁵ Compounds of the type $\text{PhC}(\text{SR})\text{:C}(\text{SR})\text{Ph}$ are cleaved by permanganate oxidation, going to benzil and a sulfonic acid.¹⁰¹⁹

Oxidation of aromatic *bis*-sulfides, $\text{ArSCH}_2\text{CH}_2\text{SAr}$ and $\text{C}_6\text{H}_4(\text{SR})_2$, gives pairs of diastereoisomeric *bis*-sulfoxides.⁹¹

Metalation of Alkyl Sulfides

Methylphenyl sulfide is metalated in the methyl group by butyl lithium: ^{486.5, 495, 1343}



When the alkyl group is larger, the lithium enters the ring.^{495, 1343} The ring is mercurated by mercuric acetate: ^{241b, 495, 1343}



Various metalating agents have been studied.^{495, 1343} The metalation of methylphenyl sulfide goes more readily than that of anisole.¹³⁴³ Diphenyl sulfide is mercurated readily.^{241b, 1129}

Sulfonium Compounds

Formation

An important reaction of aliphatic sulfides is their union with alkyl halides to form sulfonium compounds:



The products are true salts, soluble in water and highly ionized. They are called sulfonium on account of their analogy to the tetraalkyl ammonium compounds which are formed in a similar way and show similar properties:

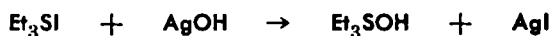


Formerly they were called sulfines. The sulfonium hydroxides, R_3SOH are strong bases resembling the tetraalkyl ammonium hydroxides, R_4NOH . There is, however, a difference in degree; sulfonium compounds are less readily formed and are less stable than the analogous ammonium compounds.^{936a}

The first sulfonium compound was made by heating ethyl sulfide and iodide together:



The product was recognized as a salt. With silver hydroxide a base was obtained:



This base gave salts when it was neutralized by various acids. The chloride formed a characteristic double salt with platinic chloride.^{952c, 952d} Similar results were obtained with methyl iodide and ethyl *i*-amyl sulfide.^{1137b} Propyl iodide unites with methyl, ethyl, and propyl sulfides. The products Me_2PrSI , Et_2PrSI , and Pr_3SI are all water-soluble salts.^{245b, 246d}

For some time chemists were concerned with the identity of the products of the two reactions: ⁷⁴⁰



The two were proved to be identical by a study of the double salts that they formed with cadmium iodide, mercuric, platinic, and auric chlorides.^{705a} Similar experiments were made with other combinations and the same conclusions reached.^{206a}

The rates of these reactions depend on the size of the alkyl groups involved. Some relative rates have been determined: ^{261a, 261b}



Methyl iodide reacts eighty-seven times as fast as ethyl iodide on methyl ethyl sulfide. Methyl iodide and sulfide combine at room temperature.¹⁰⁸² The reaction is bimolecular and is faster in the presence than in the absence of water.^{261a} The nature of the solvent has a marked effect.^{328, 901, 1098} The rates in several alcohols at 78° are as follows: ^{261a}

MeOH	50.9	PrOH	12.9
EtOH	16.5	i-PrOH	7.6

A solvent with high dielectric constant is desirable; methanol is best for purity of product.¹⁶² The formation is aided by the presence of mercuric chloride or bromide.¹⁰⁸⁵ The temperature coefficient of the reaction of methyl iodide with ethyl sulfide has been studied.¹²⁴³

Methyl iodide combines with the higher alkyl sulfides and with mixed sulfides: methyl-cyclohexyl,^{158b} methyl-octyl, methyl-dodecyl, methyl-cetyl,⁷⁴² ethyl-lauryl, ethyl-cetyl,¹⁶² and methyl-amyl,²⁷⁸ with dithiane,⁸⁶¹ dimethoxymethyl sulfide, (MeOCH₂)₂-S,⁷⁴⁷ and benzyl sulfide.⁷⁷³

In place of methyl iodide, dimethyl sulfate may be used to react with an alkyl,^{96, 592, 921, 1057, 1313} or an aryl-alkyl sulfide.^{44c, 65, 185a, 315, 540, 674, 675} An ester of a sulfonic acid may react with a sulfide.^{947c, 948a} An alkyl sulfide, a primary alcohol, and sulfuric acid, or a sulfonic acid, heated together give a sulfonium salt.^{422, 949d, 989a, 989b, 990a, 990b} An excess of a dialkyl sulfate may alkylate a mercaptan and then convert the resulting sulfide to a sulfonium salt.^{285c}

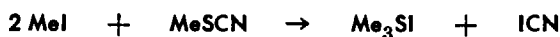
Aryl sulfonium salts, Ar₃SCl, can be prepared by more complicated reactions starting with diaryl sulfites.^{330, 331a, 331b, 775} Methyl sulfide and iodine form methyl sulfonium iodide.^{260c} Electrolysis of benzyl sulfide, in acetic-sulfuric acid solution, leads to tribenzyl sulfonium sulfate.⁴²²

Methyl iodide gives trimethyl sulfonium iodide, when heated with sodium sulfide. Stannous and cadmium sulfides, with excess methyl iodide in methanol, are transformed into the respective double salts, Me₃SI·SnI₂ and Me₃SI·CdI₂.^{705b, 705c} Methyl iodide, heated in a sealed tube with sulfur at 160 to 190°, ^{704b, 704c, 863} or with arsenic trisulfide at 100°, ^{704c} gives trimethyl sulfonium iodide.

Vulcanized rubber has been treated with methyl iodide in an

effort to get evidence of sulfide linkages. Combined sulfur should be removed as trimethylsulfonium iodide.^{1169, 1170a, 1170b}

Methyl iodide and methyl thiocyanate give trimethyl sulfonium iodide: ^{245d, 246d}



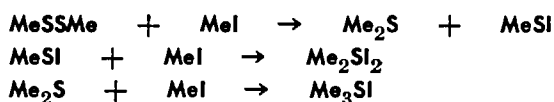
One of the products of the reaction of zinc ethyl on ethyl sulfide iodide, Et_2SI_2 , is triethylsulfonium iodide.^{1055a} Tribenzylsulfonium chloride is formed when benzyl sulfide is treated with aluminum chloride.^{755a} The colored solutions of aryl sulfides in concentrated sulfuric acid seem to contain some sort of sulfonium ions.¹²⁵⁶

Methyl and ethyl nitrates unite with alkyl sulfides on long standing. The sulfonium nitrates, so obtained, are soluble in water, methanol, and ethanol, but insoluble in benzene and acetone. Methyl formate and methyl sulfide give crystals.¹⁰⁵⁹

A sulfonium nitrite has been made from a sulfonium iodide and silver nitrite.⁵ The chlorate, Me_3SClO_3 , which has been made similarly, explodes at 170° .³⁴² The perchlorates, $\text{EtMe}_2\text{SClO}_4$, $\text{PrMe}_2\text{SClO}_4$, and $\text{BuMe}_2\text{SClO}_4$ have been compared with the alkylammonium perchlorates.⁵⁹⁸ Tribenzylsulfonium perchlorate is known.^{584c, 976c} *p*-Tolyldimethylsulfonium picrate, perchlorate, and bichromate have been prepared.⁶⁷⁵ Trialkylsulfonium picrates have been made.¹⁰¹³ The melting points of a number of these have been recorded.^{646, 1313} The fact that so many of the melting points are between 67 and 76° suggests that they may be decomposition temperatures rather than melting points.

Hydroxyethyl-methyl sulfide, $\text{HOCH}_2\text{CH}_2\text{SMe}$, and thiodiglycol, $\text{S}(\text{CH}_2\text{CH}_2\text{OH})_2$, combine readily with alkyl halides to form sulfonium salts. This will be discussed more fully when the hydrolysis of mustard gas is considered.^{191, 224, 285a}

Alkyl disulfides, as well as sulfides, give sulfonium salts with alkyl halides.^{299, 581, 1226, 1408} The reaction is speeded up by mercuric iodide.⁵⁸¹ The reactions seem to be: ¹²²⁶



Some α -halogen-ketones add to alkyl sulfides: ^{60.5, 1197b}



Phenacyl^{140, 738, 1126, 1288} and *p*-phenylphenacyl bromides react in this manner.¹⁶³ An α -chloromethyl sulfide combines with methyl sulfide:¹³⁸



Acetyl chloride reacts with ethyl sulfide, but the constitution of the product is not clear.¹¹³⁹ Methyl iodide reacts with alkyl phenacyl sulfides about ten times as fast as ethyl iodide.¹⁰²⁸

Methyl,^{218a, 218b, 765, 766} ethyl,^{218a, 260b, 262b, 378, 765, 766} propyl,⁷⁶⁵ *i*-butyl,⁷⁶⁵ *i*-amyl,⁷⁶⁵ and methyl-ethyl^{260b, 262b} sulfides have been combined with bromoacetic acid, methyl-dodecyl sulfide with chloroacetic acid,¹³⁵⁵ and methyl,^{114, 260b, 262b} ethyl,¹¹⁴ and methyl-ethyl sulfides¹³¹⁵ with α -bromopropionic acid to make sulfonium compounds. The kinetics of the reaction have been studied with iodoacetic acid and methylmercapto- and ethylmercaptoacetic and thiodiglycolic acid.⁵⁶⁴ β -Propiolactone forms sulfonium salts with alkyl sulfides.^{125.5, 1111} The biological decomposition of the salts, $\text{R}_2\text{S}(\text{Cl})\text{CH}_2\text{CH}_2\text{CO}_2\text{H}$, has been studied.²⁷⁵

Double sulfonium salts have been prepared from methyl sulfide and ethylene bromide^{245a, 245c, 246a, 246b, 351, 861} and from bis(ethyl-mercapto)ethane, $\text{EtSCH}_2\text{CH}_2\text{SEt}$, and bis(allylmercapto) ethane²³⁶ with methyl iodide.^{189, 1345}

Resolutions of sulfonium compounds have been effected. Methyl-ethyl-carboxymethyl sulfonium bromide and silver *D*-camphor sulfinatate gave a salt that was resolved.¹⁰¹⁶ Methyl-ethyl sulfide and *D*-menthyl bromoacetate combined to give such a salt directly.^{1197c} From a physicochemical study, it has been concluded that racemization of a sulfonium salt takes place through dissociation and recombination:⁶⁹¹



Characteristics

It has been claimed that trialkylsulfonium compounds, R_3SX , exist in two isomeric forms, the usual ionizable which is colorless and a colored nonionizable.⁵⁴¹

The refractivity of trialkylsulfonium compounds has been studied.^{936b} Triethylsulfonium iodide is rhombic.⁹²⁵ At 25°, 431 g. of this iodide dissolves in 100 g. of water and 47.7 g. in the same weight of chloroform.⁹⁸² The crystal forms of the trimethylsulfonium chloride, bromide, and iodide could not be measured

on account of instability,⁹²⁵ but the double salts with tin chloride are stable.³²⁷

The Raman spectrum for trimethylsulfonium bromide in water solution has been determined. The ion has a pyramidal structure with the sulfur atom at the apex.¹¹⁸⁷ The infrared spectra of a number of sulfonium compounds have been studied.^{328, 1272, 1294} The mobilities,^{97, 505} bond distances, and activation energies⁶⁹¹ have been estimated for the ions, R_3S^+ .

The electrolytic dissociation of trialkylsulfonium salts in a variety of solvents has been determined.^{59, 262c, 1308}

As the trialkylsulfonium salts are soluble in water and insoluble in hydrocarbons, alkyl halides may be separated from them by the use of water and a low-boiling hydrocarbon.^{947b}

Reactions

Complexes with Metal Salts

Trialkylsulfonium halides give complexes with salts of platinum,^{158b, 158c, 191, 218a, 218b, 245b, 245c, 246b, 246d, 260b, 262a, 262b, 341, 347, 351, 599, 674, 700c, 704b, 704c, 705a, 705c, 740, 765, 861, 937b, 952b, 952d, 1137b, 1150, 1197c, 1236a, 1236b, 1315, 1345} gold,^{218, 674, 705a, 740, 765, 861} mercury,^{68, 331a, 331b, 341, 524, 705a, 740, 861, 1061a, 1068, 1085, 1236a, 1236b, 1345, 1348} cadmium,^{123, 341, 705a, 705c, 1062, 1236a, 1236b, 1345, 1348} copper,^{123, 343, 344} tin,^{123, 351, 705c, 1236a, 1236b} zinc,^{123, 1062, 1348} manganese,¹²³ iron,^{123, 524, 599} antimony,^{1064a} bismuth,^{123, 657, 733, 1057, 1314} thallium,⁶⁵⁷ silver,^{477a, 978a, 978c} and lead.⁷⁶⁵ Triphenylsulfonium bromide is used in testing for bismuth and cobalt.¹⁰²¹ Trimethylsulfonium fluoride, $Me_3SF \cdot H_2O$, and silicofluoride, $(Me_3S)_2SiF_6$, have been described.³⁹⁶

Triethylsulfonium sulfate forms double sulfates with ferrous, zinc, cobalt, cadmium, magnesium, manganese, nickel, and copper sulfates.¹⁰⁷² Tribenzylsulfonium chloride forms a double salt with ferric chloride.⁵⁹⁹ Trimethylsulfonium chromate and bichromate form double salts with mercuric cyanide.^{1236c}

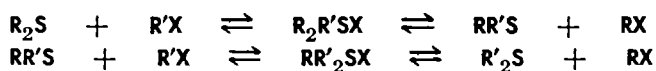
Ethyl sulfide displaces the ether from triethyloxonium borofluoride to form triethylsulfonium borofluoride, Et_3SBF_4 .^{881, 882} Iodoform gives crystalline addition compounds with sulfonium salts: ^{405, 1227} $Et_3S(OH) \cdot CHI_3$, m. 126° , $Et_3SCl \cdot CHI_3$, m. 96° , $Et_3SBr \cdot CHI_3$, m. 124° , $Et_3SI \cdot CHI_3$, m. 142° ,⁴⁰⁵ and $EtMe_2SI \cdot CHI_3$, m. 136° .^{405, 1227}

Decomposition and Radical Exchange

The addition of an alkyl halide to a sulfide is a reversible reaction:



The sulfonium salt is in equilibrium with its components under all conditions. Pressure naturally forces the equilibrium to the right while an elevation of the temperature has the opposite effect. When two or more different alkyl radicals are involved the products of the dissociation may be different from the original components:



At equilibrium, there will be two sulfonium salts, three sulfides, R_2S , RSR' , and R'_2S , and two halides $R'X$ and RX , or seven compounds in all. If there are three different alkyl groups the situation will be even more complex. The maintenance of the equilibrium depends on all of these compounds remaining within the sphere of action. In case any one of the products is eliminated, the equilibrium will be displaced. If there is an excess of $R'X$ and RX is volatile or unstable R_2S will disappear and the final product will be R'_3SX .

Methyl *p*-cresyl sulfide unites with dimethyl sulfate. On heating, the product dissociates into its components: ^{44c}



When methyl *p*-cresyl sulfide is heated with chloroacetic acid, 80% of $p\text{-MeC}_6\text{H}_4\text{SCH}_2\text{CO}_2\text{H}$ is produced, indicating the formation and decomposition of a sulfonium chloride. The volatile methyl chloride escapes.⁷³⁹ There may be a sort of disproportionation: ⁷⁶⁵



β -Benzylmercaptopropionic acid, $PhCH_2SCH_2CH_2CO_2H$, heated with dimethyl sulfate and alkali, gives methyl benzyl sulfide, indicating the formation and decomposition of a sulfonium salt.^{602c}

Cyclic trithioformaldehyde, when heated with methyl iodide and methanol, gives up to 90% of trimethyl sulfonium iodide and

methylal, indicating formation and methanolysis of the cyclic sulfonium compound.¹⁰⁹⁰ Trithioacetaldehyde and methyl iodide unite to form a sulfonium compound which decomposes at 215° without melting. The products are trimethylsulfonium iodide and ICHMeSCHMeSCHMeI .^{704a} Sulfonium iodides are obtained from mono- and dithiopaldehydes with methyl and ethyl iodides.¹⁰⁰⁵ Cyclic sulfides, $(\text{CH}_2)_n > \text{S}$, are decomposed by methyl iodide into trimethylsulfonium iodide and the diiodide, $\text{I}(\text{CH}_2)_n\text{I}$.⁹¹⁸

Cyclohexyldimethyl sulfonium iodide is decomposed by alcoholic potash into methyl sulfide and cyclohexene.^{1349b}

An early chemist was surprised to find trimethylsulfonium iodide, Me_3SI , as the product when he treated secondary butyl sulfide, $s\text{-Bu}_2\text{S}$, with methyl iodide.¹⁰⁹¹ The s -butyl iodide is less volatile than the methyl iodide, but is far less stable. Triethylsulfonium iodide can be converted to the trimethyl compound by heating with methanol. The reverse takes place when the trimethylsulfonium iodide is heated with excess ethanol.^{705b} A variety of products may result from the action of an alkyl halide on a mixed sulfide.¹⁰⁵⁸ In the chapter on cyclic sulfides, examples will be given of the conversion of linear polymers into rings.

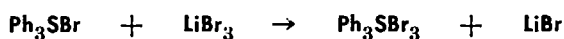
The decomposition of triethylsulfonium bromide in a number of solvents has been investigated. The reaction is of the first order; its rate depends on the solvent.^{328, 531, 532, 1098}

Oxidation and Reduction

Oxidation of a trialkyl sulfonium compound gives a sulfonic acid, or a sulfone, or both.^{218b, 766} Triethylsulfonium hydroxide is reduced to ethyl sulfide, ethane, and water.¹⁶

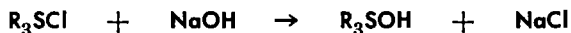
Halogenation

A trimethylsulfonium halide takes up two atoms of a halogen, the original halogen apparently becoming trivalent. The following compounds have been identified: Me_3SI_3 , $\text{Me}_3\text{SI}_2\text{Br}$, $\text{Me}_3\text{SI}_2\text{Cl}$, Me_3SIBr_2 , Me_3SIBrCl , Me_3SICl_2 , Me_3SBr_3 , $\text{Me}_3\text{SBr}_2\text{Cl}$, Me_3SCl_3 . The first and last of these are unstable.³⁶⁸ Trimethylsulfonium iodide, chlorinated in acetic acid solution, gives the complex, $\text{Me}_3\text{SCl} \cdot \text{ICl}_3$, which gives off chlorine when heated.¹³⁵⁴ Triphenylsulfonium bromide takes up bromine from bromine water. In an evacuated tube, bromine migrates from lithium tribromide: ¹⁵²



Trialkylsulfonium Hydroxides, R_3SOH

As mentioned earlier, the hydroxide may be prepared from a halide and silver hydroxide. Another way is to add sodium hydroxide dissolved in absolute alcohol to a like solution of a trialkylsulfonium chloride:

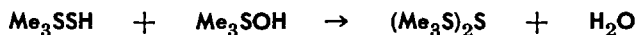


Sodium chloride, being nearly insoluble in the alcohol, precipitates out.⁷⁷⁶

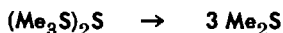
Triethylsulfonium hydroxide behaves like sodium hydroxide as regards change of conductivity with dilution.^{201, 959} With carbon disulfide and an alcohol, it gives the xanthate reaction:⁴¹⁸



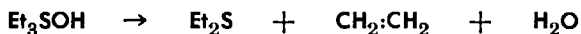
Toward hydrogen sulfide, trimethylsulfonium hydroxide acts like sodium hydroxide. The hydrosulfide reacts with more of the base to form the sulfide:



This decomposes in aqueous solution:



The hydrosulfide is oxidised by air.^{124, 217} With aluminum, the triethyl hydroxide gives the basic aluminum compound, Et_3SOAlO . On heating, the triethyl hydroxide decomposes into ethyl sulfide, ethylene and water:⁵¹³



In solutions, so dilute that the dissociation of a trialkylsulfonium hydroxide may be considered as complete, the decomposition reaction is of the second order, the velocity being proportional to the concentration of each of the ions, R_3S^+ and OH^- .⁵¹³ The primary decomposition may be thought of as giving the sulfide and the alcohol:



Dehydration of the alcohol to the olefin and water may be regarded as a secondary reaction. Actually, olefin and alcohol are produced, their relative amounts depending on the nature of the

alkyl groups present and the conditions of the decomposition. This is particularly true when alkyls of different nature are present. This decomposition has been thoroughly investigated recently.^{322, 323, 324, 501, 614, 615, 616, 617, 618, 645, 646} Methyl vinyl sulfide is obtained by the decomposition of a sulfide-sulfonium base:³³⁴



The sulfonium iodide from dithiane and methyl iodide, with silver hydroxide, gives silver iodide and a compound, $\text{C}_5\text{H}_{10}\text{S}_2$, boiling at 210 to 212°, which is probably $\text{MeSCH}_2\text{CH}_2\text{SCH:CH}_2$. Boiling with sodium hydroxide yields the same product.^{893b}

Uses

Certain sulfonium compounds are claimed as useful in dyeing or in otherwise treating textiles.^{65, 300, 315, 630c, 644, 921} Those containing one or more high molecular weight radicals may serve as foaming, wetting, dispersing, or emulsifying agents.^{142, 143, 299, 393, 948a, 949d, 989b, 990a} An octyldimethylsulfonium salt is claimed as a photographic sensitizer.²⁶⁵ Lubricants are said to be improved by the addition of trialkylsulfonium salts.⁹¹² Linear polymeric and high molecular weight sulfonium compounds have excited some interest.^{549a, 1151, 1166, 1283} Sulfonium salts containing special groups are recommended as disinfectants and fungicides.^{285b, 540, 742, 942} Polysulfonium compounds derived from linear polymeric sulfides are said to be useful in the treatment of textiles.^{316a}

Substituting sulfur for nitrogen in compounds of the betaine and choline types diminishes the physiological effects.^{621, 868, 1029} It has been suggested that the ethyl sulfide formed in the intestine by the decomposition of cystine is detoxified by methylation to the sulfonium compound. Methyl-diethylsulfonium hydroxide has been identified in dog urine.⁹⁴¹

Selenonium Salts

Alkyl selenides combine in a similar way with alkyl halides:¹⁹⁸



The base Et_3SeOH has been prepared from the iodide and silver hydroxide. Many of its salts are unstable.¹⁰⁰² Methyl phenyl selenide and dimethyl sulfate combine.⁶¹ When this selenide is

heated with bromoacetic acid, phenyl selenoacetic acid, $\text{PhSe-CH}_2\text{COOH}$, is formed, proving the presence of a selenonium intermediate.³⁸⁹ Aromatic selenonium salts may be formed in quite a different way. Diphenyl selenium dichloride reacts with benzene, in the presence of aluminum chloride, to form triphenylselenonium chloride, Ph_3SeCl , which is a salt. On heating, this decomposes into phenyl selenide and chlorobenzene.⁷⁵⁸ Selenonium compounds with three unlike radicals have been resolved.¹⁰¹⁵ An x-ray study has been made of triphenylselenonium chloride.⁸²⁹

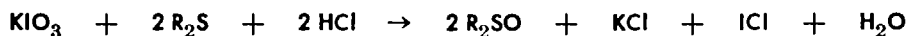
Detection and Estimation of Sulfides

This subject has been touched upon in Chapter 2 of Volume I, since alkyl sulfides are commonly present with mercaptans in petroleum distillates. The alkyl sulfides in petroleum products are estimated "by difference," that is the sulfur not otherwise accounted for is considered to be present in sulfides. The methods have been reviewed.^{310, 667.5} In testing for sulfides and in determining their amount, the presence of other classes of sulfur compounds must be taken into account. The more reactive mercaptans and disulfides, which are particularly confusing, must be eliminated.

Hexanitrate-ammonium cerate gives a sensitive color test which appears to be generally applicable to alkyl sulfides. Thiophenes do not interfere but alcohols do.⁵⁵¹ Sodium iodoplatinate gives a blue color with 35 gamma of butyl sulfide and with comparable amounts of other sulfides.⁶²⁸

The mass spectrograph may be used with sulfides as with other sulfur compounds.^{414, 1368} Chromatographic methods are being adapted to the separation and determination of alkyl sulfides.^{414, 627, 762} Silica gel is a particularly good adsorbent.⁵⁴³

The lamp,¹³⁴² Parr bomb, and oxygen bomb methods are applicable to alkyl sulfides as well as to other sulfur compounds. Oxidation to the sulfate ion by chloric, or nitric, acid does not go as well with sulfides as with other organic sulfur compounds,¹²⁵⁵ since the sulfones are exceptionally stable. A potentiometric titration may be employed.⁸¹⁰



Advantage may be taken of the fact that silver nitrate forms a complex with a sulfide. A measured amount of standard silver

nitrate is added to a solution containing a sulfide and the excess titrated with thiocyanate.⁵¹⁹

Mercurous nitrate may be regarded as in equilibrium with the mercuric salt and free mercury:

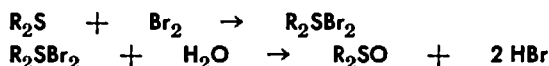


The addition of a sulfide, which can combine with mercuric nitrate but not with mercurous nitrate, shifts this equilibrium to the right:



The mercury may be collected and weighed.^{404b, 1108} Thiophene interferes.⁹³²

The formation and hydrolysis of the bromine addition product can be used for the estimation of an alkyl sulfide:



Both the bromine used up and the hydrobromic acid formed are titrated. Excess of the first over the second indicates the presence of unsaturates.^{94, 610.5, 1134, 1167.5} If substitution has taken place, the acid is low. Phenyl benzyl sulfide has been determined by measuring the bromine consumption in *t*-butanol.^{547b} A bromide-bromate solution may be used for the titration.^{414, 1189}

Concentrated sulfuric acid has been used to absorb ethyl sulfide from a current of dry air. The acid is chilled and diluted with water and standard iodine solution added, keeping the temperature at 0°. After this stands for some hours, the excess iodine is titrated.²⁸⁷

Monosulfide links in coal¹⁰²⁰ and in vulcanized rubber⁸⁹¹ have been determined by the addition of methyl iodide.

An instrument has been devised for the continuous titration of sulfides in a gas stream.⁴⁰

Physiological Effects of Sulfides

Physiological effects of methyl sulfide have been reported.^{787, 1107} It is only mildly toxic, a concentration of 5% by volume being required to kill rats in 15 minutes.⁷⁸⁷ One percent of ethyl sulfide in anesthetic ether produces severe gastroenteritis.¹⁷⁴ Allyl sulfide has been investigated more extensively. It is more active than

the saturated sulfides.^{1099, 1262} It acts on the respiratory center of the medulla.²⁵⁴ Gapeworms in chicks can be eliminated by it.³⁰⁴ The sulfur in the urine of a pig rises after its administration.⁹⁹⁴ Allyl sulfide has antiseptic properties¹³¹⁰ and has been found to be beneficial for accessible tuberculosis lesions.³⁹⁴ The sulfides PhSEt and *p*-RSC₆H₄C(:NH)NH₂ in which R is Me, Et or Pr, have been tested against tuberculosis bacillus and against pneumococcus.²⁴⁰ The sulfides *m*-O₂NC₆H₄SR in which R is Me, Et or Am, are anticoccidial agents and useful in poultry feed.¹³³⁸ The polyvinyl sulfides are strong bactericidal agents.¹⁴¹⁵

Several aromatic sulfides,¹²⁹⁸ nitrophenyl, nitrophenyl nitrothienyl, and bis(nitrothienyl) sulfides have been tested for antibacterial efficiency.³⁴⁰ Phenyl sulfide and several of its methyl and halogen substitution products are synergistic with nicotine in insecticides.¹³⁸⁵ The hydroxyphenyl sulfides have been compared with the corresponding ethers.⁶⁹⁹

Industrial Uses of Sulfides

Without any pretense of completeness, a few references are given as to uses. Various sulfides and mixtures containing them have been claimed as insecticides,^{225, 406, 575, 634b, 790, 907, 1111} fungicides,^{406, 631b, 1270} fumigants,^{337, 635, 791, 939, 1105} and disinfectants.^{629a, 1351} The double sulfide, PhSCH₂CH₂SPh, is mildly toxic to culicine mosquito larvae.²⁵⁰

Methyl sulfide and ethyl mercaptan are powerful attractants for female blowflies.³³³

The toxicities of different classes of sulfur compounds to platinum and nickel hydrogenation catalysts have been compared.^{869, 870} Methyl^{871, 1101} and ethyl sulfides^{1160, 1389} are catalyst poisons. Alkyl sulfides are antagonistic to lead tetraethyl but less so than disulphides or mercaptans.⁷⁸⁶

Certain sulfides are said to be useful in vulcanization accelerators,^{320, 474, 637b, 887, 1087a, 1312} others in soaps and creams,^{565b} others in textile treatments,^{225, 549b, 637b, 779, 1087a} and others as constituents of rubberlike polymers^{384a, 1217, 1218} or of resins.^{821a} Phenyl sulfide, used in high-temperature aqueous digestion of wood, improved its plastic properties.⁵³³

Aliphatic sulfides are thermal stabilizers in polymethyl methacrylate polymers.⁸⁴⁶ Several are used in emulsion polymerization.⁵¹¹ Ethyl phenyl sulfide stabilizes polysulfone resins.^{335.5}

Higher alkyl sulfides, such as dilauryl sulfides, are claimed as emulsifying and flotation agents.¹¹³³ High molecular weight sulfides having a carboxyl or sulfonic group at one end are said to be useful as surface-active agents.^{565a}

Methyl, ethyl, and propyl sulfides in 1% concentration cut down the oxidation of furfural in oxygen by 97%.⁹¹⁴ They diminish the autooxidation of amines, but only slightly.¹³³⁰ Small amounts, 5 to 100 parts per million, keep down the oxidation of methanol to formaldehyde.⁹⁸¹ Substituted diphenylsulfides are said to be age resisting in rubber compounds.⁶⁴³ Sulfides have been found useful in stabilizing transformer and lubricating oils.^{232, 235, 249, 309, 356a, 358, 672, 736, 808, 900, 1114, 1375} The film strength^{309, 311, 690, 992, 1039, 1335} and other properties of lubricating oils^{256, 348b, 399, 826, 922, 1083, 1135, 1168, 1323, 1403} are improved by the addition of certain sulfides. Allyl 3-thienyl sulfide is claimed as an oil additive.^{210b} The tris-sulfide, $S(CH_2CH_2SAm)_2$, is said to be a noncorrosive lubricant for instruments.⁷⁶ The addition of 0.1 to 5% of a sulfide to the fuel in a gas turbine is said to be beneficial.¹³²² Alkyl selenides are said to be particularly effective as antioxidants in oils.^{249, 356b, 357, 1232.5}

CHAPTER 3.

Physical Properties of Sulfides

Introduction

The properties of a sulfide are of importance, in the first place, for its identification. When a chemist prepares a new compound, he must determine and record its properties so that it may be identified. When a number of sulfides have been prepared and characterized, it becomes interesting to compare the properties in order to find the relationships to the structures and molecular weights.

The purpose of the tables and property lists is to give information about the properties of sulfides. In addition, the data on each compound show the state of our knowledge and, by reference to the bibliography, will direct the reader to the original articles.

The theme that runs all through this book is the analogy between sulfur and oxygen compounds. In Volume I, the analogies between alcohols and mercaptans were traced. In this chapter sulfides are compared with ethers. The resemblance of sulfides to ethers is closer than that of mercaptans to alcohols.

Comparison of Properties

In this section the properties of some selected sulfides are discussed. Particular attention has been given to their melting points which are compared with those of hydrocarbons and ethers.

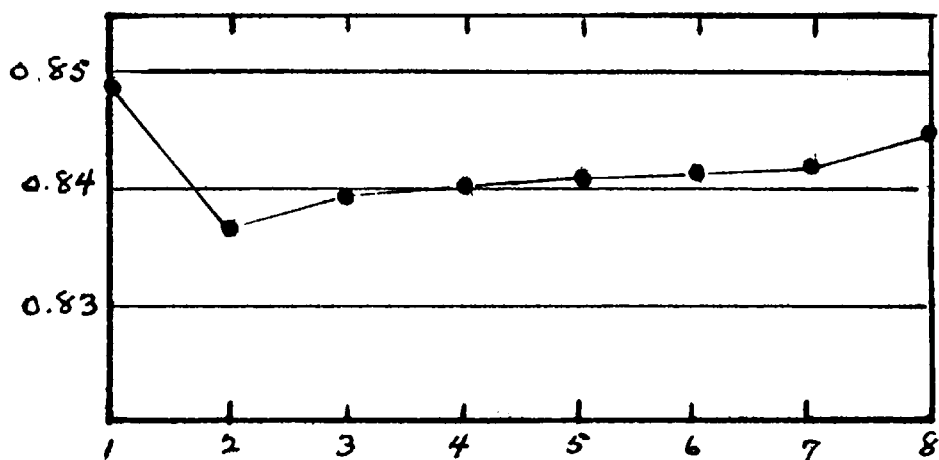
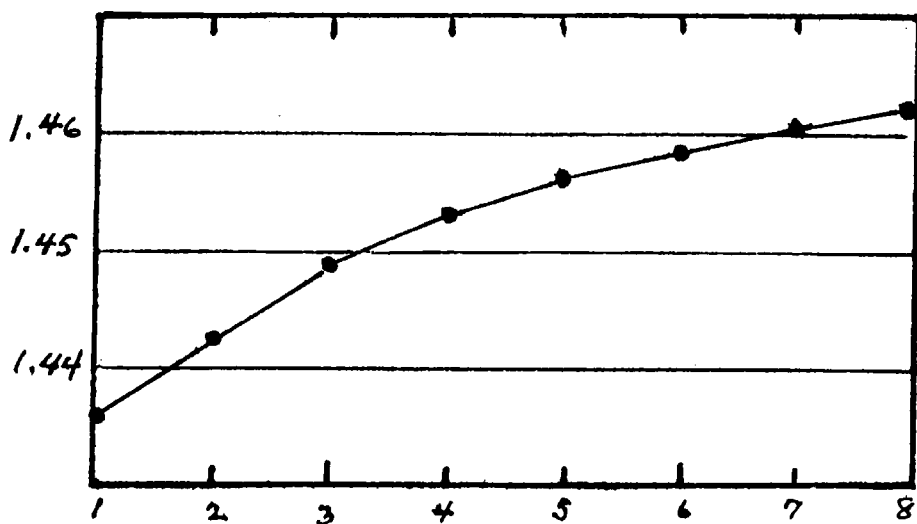
In Table 1.3 selected values for several properties of the simpler aliphatic sulfides are brought together.

TABLE 1.3
Comparison of Properties of Some Alkyl Sulfides

Formula	M.p., °C	B.p., °C	d 20/4	N 20/D	σ 20°	Parachor
Me ₂ S	—98.25° ^a	37.3° ^a	0.8483 ^a	1.4353 ^a	24.44	163
MeSEt	—105.91° ^a	66.6° ^a	0.8422 ^a	1.4403	25.16	201.8
EtSEt	—103.01° ^a	92.0°	0.8363 ^a	1.4427 ^a	25.13	241.1
MeSPr	—112.98 ^a	95.6° ^a	0.8424 ^a	1.4442 ^a		
MeSBu		122.5°	0.8427	1.4478	26.25	280.1
EtSPr	—117.04° ^a	118.5° ^a	0.8370 ^a	1.4461 ^a		
EtSBu	—95.13° ^a	144.2° ^a	0.8376 ^a	1.4491 ^a	26.49	320.5
PrSPr	—102.6° ^a	142.8° ^a	0.8377 ^a	1.4487 ^a	26.34	319.1
<i>i</i> -Pr ₂ S	—78.08° ^a	119°	0.8166	1.4395	23.20	317.6
BuSBu	—79.7° ^b	187°	0.8402	1.4530	27.24	397.7
<i>i</i> -Bu ₂ S	—105.5° ^c	169°	0.8263	1.4469	24.95	395.5
<i>s</i> -Bu ₂ S		165.5°	0.8348	1.4506	25.87	395.4
Am ₂ S	—51.3° ^d	227.4° ^e	0.8409	1.4562	27.81	476.0
<i>i</i> -Am ₂ S		216° ^{f,g}	0.8340	1.4527	26.04	472.1
Hex ₂ S	—28.0° [*]	230° ^h	0.8411	1.4586	28.52	556.0
Hep ₂ S	—10.0° [*]	298° ⁱ	0.8416	1.4605	29.29	637.2
Oct ₂ S	4° [*]	310° ^k	0.8447	1.4621	30.43	719.2
<i>i</i> -PrSMe	—101.48° ^a	84.8° ^a	0.8291 ^a	1.4392 ^a		
<i>i</i> -PrSEt	—122.19° ^a	107.3° ^a	0.8246 ^a	1.4407 ^a		
<i>i</i> -PrSPr		132.1° ^a	0.8269 ^a	1.4440 ^a		
<i>i</i> -BuSMe		112.5°	0.8335	1.4433	24.99	229.6
<i>i</i> -BuSEt		134.2° ^a	0.8306 ^a	1.4450 ^a		
<i>s</i> -BuSEt		133.6° ^a	0.8353 ^a	1.4477 ^a		
<i>t</i> -BuSMe		99°	0.8257	1.4402	23.49	277.9
<i>t</i> -BuSEt	—88.95 ^a	57°/109	0.8200	1.4416	23.50	317.5

* Estimated. Unmarked data from Vogel and Cowan (1326). a McAllan, Cullum, Dean, and Fidlers (819), b 1291b, c 1293, d 1192, e 46a, f 64, g 818a, h 1367, i 984, j 1376b, k 902.

The densities, as seen in Figure 1.3, go down from methyl to ethyl and then rise slowly. The refractive indices, which are plotted in Figure 2.3, go up slowly.

FIGURE 1.3. *Density, $d_{20/4}$, of the Sulfides*FIGURE 2.3. *Refractive Indices, $n_{20/D}$, of the Sulfides*

In Figure 3.3, the boiling points are plotted against the molecular weights, along with those of the normal paraffins, ethers, and alkyl disulfides. The curve for the sulfides is slightly higher and that for the ethers is slightly lower than the one for the hydrocarbons. The curve for the disulfides is only a little above that for the sulfides.

From these curves the molecular weights of the hypothetical hydrocarbons having the same boiling points as the sulfides have been estimated. These are given in Table 2.3 along with those

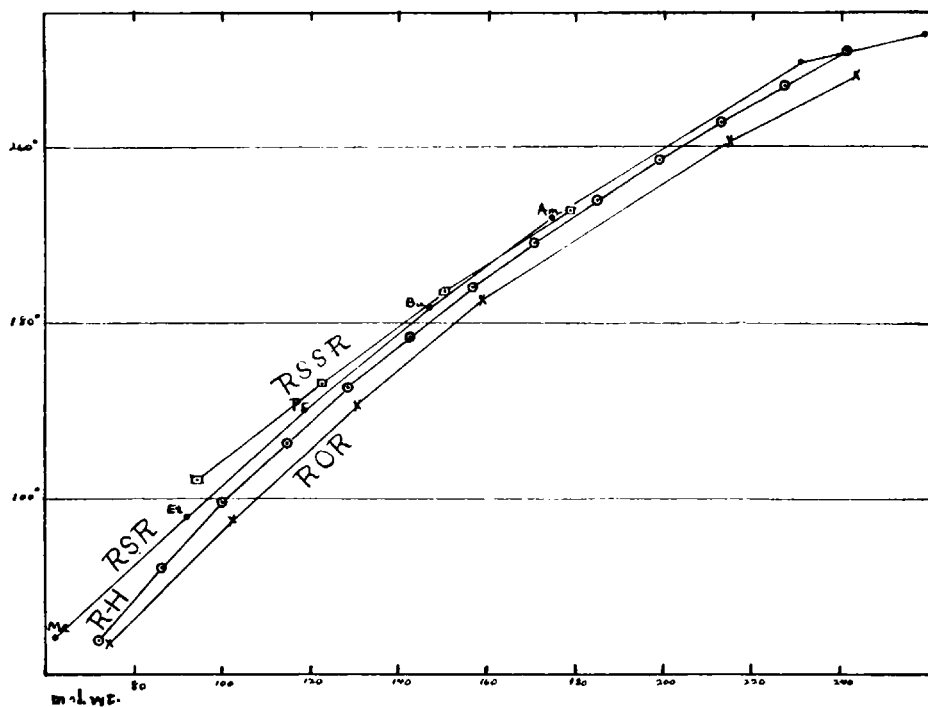


FIGURE 3.3. Boiling Points of Sulfides, RSR, Disulfides, RSSR, Ethers, ROR, and Hydrocarbons, RH, Plotted against Molecular Weights

of the sulfides. The ratios of the two sets of figures appear to indicate some association of the lower sulfides.

TABLE 2.3

Molecular Weights of Sulfides and of Hypothetical Hydrocarbons Having the Same Boiling Points

	Methyl	Ethyl	Propyl	Butyl	Amyl	Heptyl
Sulfide	62.1	90.2	118.2	146.3	174.3	230.4
Hydrocarbon	73.9	97.5	122.7	150.9	178.2	236.2
Ratio	1.19	1.08	1.04	1.03	1.022	1.021

As is shown in Table 3.3, the alkyl sulfides, except methyl, boil somewhat lower than the isomeric mercaptans. In any group of isomeric sulfides the most symmetrical sulfide boils at the lowest temperature.

TABLE 3.3
Boiling Points of Sulfides and Isomeric Mercaptans

Sulfide	B.p., °C	Mercaptan	B.p., °C	Difference
Dimethyl	37.3°	Ethyl	34.7°	-2.8°
Methyl Ethyl	66.6°	Propyl	68°	1.4°
Diethyl	92.0°	Butyl	98°	6°
Methyl Propyl	95.6°			
Ethyl Propyl	118.5°	Amyl	126.5°	8°
Methyl Butyl	122.5°			
Dipropyl	142.8°	Hexyl	151.5°	8.7°
Dibutyl	187°	Octyl	198.1°	12°

In Table 4.3, the melting points of the symmetrical sulfides, RSR, are contrasted with those of the ethers, ROR, and with those of the hydrocarbons, R·R, having the same number of carbon atoms. These are plotted in Figure 4.3.

TABLE 4.3
Melting Points (°C) of Hydrocarbons, Sulfides, and Ethers

R	R·R	Difference	RSR	Difference	ROR	RS·SR
1	-182.3°	-84.0°	-98.3°	40.2°	-138.5°	-84.7°
2	-138.5°	-32.6°	-105.9°	10.4°	-116.3°	-101.4°
3	-95.3°	7.3°	-102.6°	19.4°	-122.0°	-85.6°
4	-56.8°	22.9°	-79.7°	18.3°	-98.0°	
5	-29.7°	21.6°	-51.3°	17.7°	-69.0°	
6	-9.6°	18.4°	-28.0°	16°	-(44°)	
7	6.2°	16.2°	-(10.0°)	14°	-(24°)	
8	18.1°	14.1°	(4°)	12°	-8°	
9	28.0°	11.0°	(17°)	12°	(5°)	
10	36.6°	9.6°	27.0°	11°	16.0°	
11	44.0°	9.2°	34.8°	10.8°	(24°)	
12	50.7°	8.5°	42.2°	9.7°	32.5°	34.5°
13	56.4°	8.2°	48.2°	9.2°	(39°)	44°
14	61.4°	7.6°	53.8°	10.3°	43.5°	46°
15	65.8°	6.8°	59.0°	8.0°	(51°)	
16	69.8°	6.8°	63.0°	8.0°	55°	55.5°
17	73.2°	7.0°	66.2°	7.2°	(59°)	60°
18	76.0°	7.0°	69.0°	7.0°	(62°)	62.5°

The data for the lower sulfides are from Table 1.3, those for the higher are from a recent investigation.¹⁰⁸⁰ The melting points of the hydrocarbons up to eicosane are the selected values published

by the Bureau of Standards. Some of the higher ones were selected by Francis. Interpolated values are in parentheses. The melting points of some disulfides have been added. They are close to those of the ethers.

Except for the first three members of these series, the melting points of the ethers are below those of the sulfides by approximately the same amount as the melting points of the sulfides are below those of the hydrocarbons of the same carbon content. Thus dibutyl ether melts 18.3° below dibutyl sulfide which melts 22.9° below *n*-octane. These differences become smaller and also more regular as the alkyl groups lengthen until they go down to 7° .

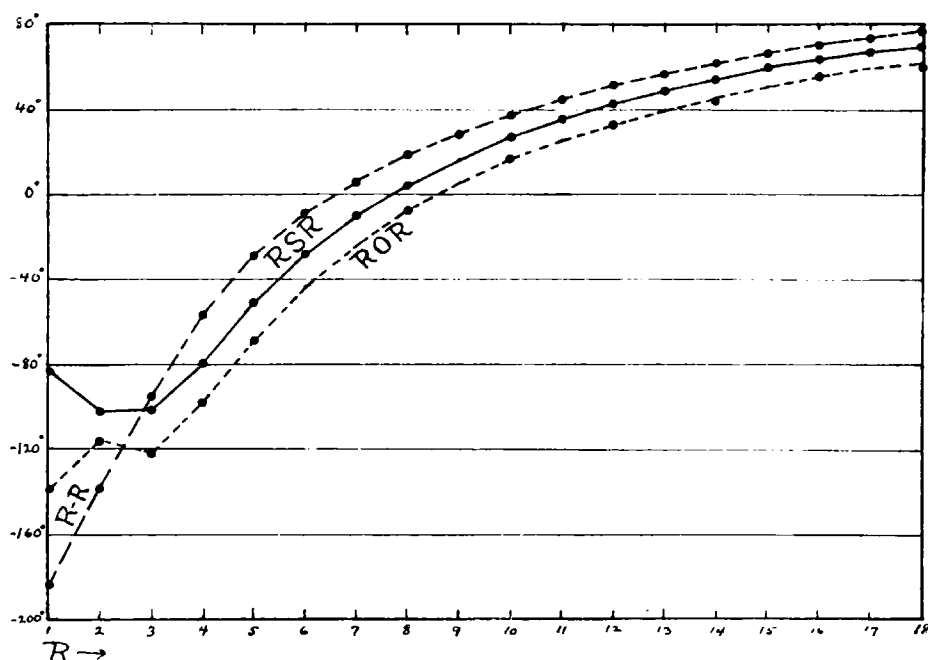


FIGURE 4.3. *Melting Points of the Sulfides, RSR, Ethers, ROR, and Hydrocarbons, RR*

In Tables 5.3 and 6.3 are listed the melting points of several series of aryl-alkyl sulfides and of the corresponding ethers. The differences between the melting points of PhSR and PhOR are nearly the same as between the alkyl sulfides and ethers, RSR and ROR , having the same alkyl radicals. Between the melting points of $p\text{-BrC}_6\text{H}_4\text{SR}$ and $p\text{-BrC}_6\text{H}_4\text{OR}$ the differences are slightly larger and show some alternation. The differences between $p\text{-MeC}_6\text{H}_4\text{SR}$ and $p\text{-MeC}_6\text{H}_4\text{OR}$ and between

β - $C_{10}H_7SR$ and β - $C_{10}H_7OR$ are smaller and show decided alternation. When R contains an odd number of carbon atoms, β - $C_{10}H_7SR$ melts lower than the corresponding ether. With the biphenyl derivatives the difference starts at about 11° where R is amyl and decreases to about 8° . Several of these series are plotted in Figure 5.3. It is remarkable that these differences are so constant in spite of the alternation. The sulfides, RSR , melt below the hydrocarbons $R \cdot R$, but the sulfides $ArSR$ melt above the hydrocarbons ArR . Thus cetyl phenyl sulfide melts at 50.8° while 1-phenyl hexadecane melts at 27° , that is, 23.8° lower. Heptadecyl phenyl sulfide melts at 57.6° , which is 19.6° above the melting point of the hydrocarbon.

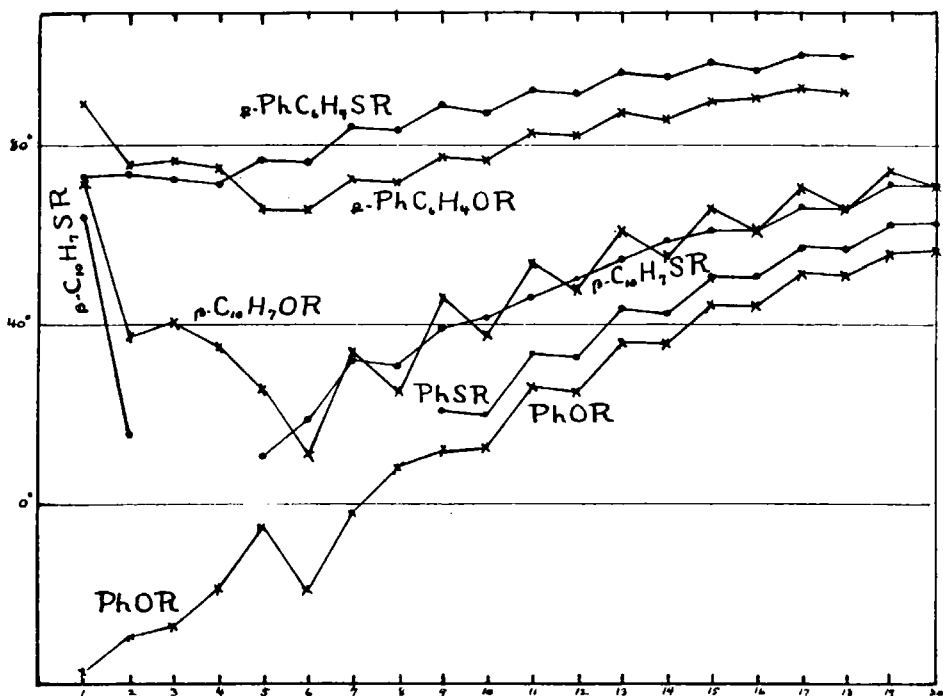


FIGURE 5.3. *Melting Points of Sulfides and Corresponding Ethers*

The melting points of the *bis*-ether sulfides, $ROCH_2CH_2SCH_2CH_2OR$, which show alternation, are plotted in Figure 6.3 along with those of the *tris*-sulfides, $RSCH_2CH_2SCH_2CH_2SR$, which do not show alternation. The *tris*-sulfides melt considerably higher than the *bis*-ether sulfides, as would be expected. The differences show alternation since the *bis*-ether sulfides show

marked alternation as is shown in Figure 6.3. When R is 10, 12, 14, 16, or 18 the lowering due to the substitution of two ether linkages for two of the sulfur is just about half the difference between sulfides and ethers for the same values of R.

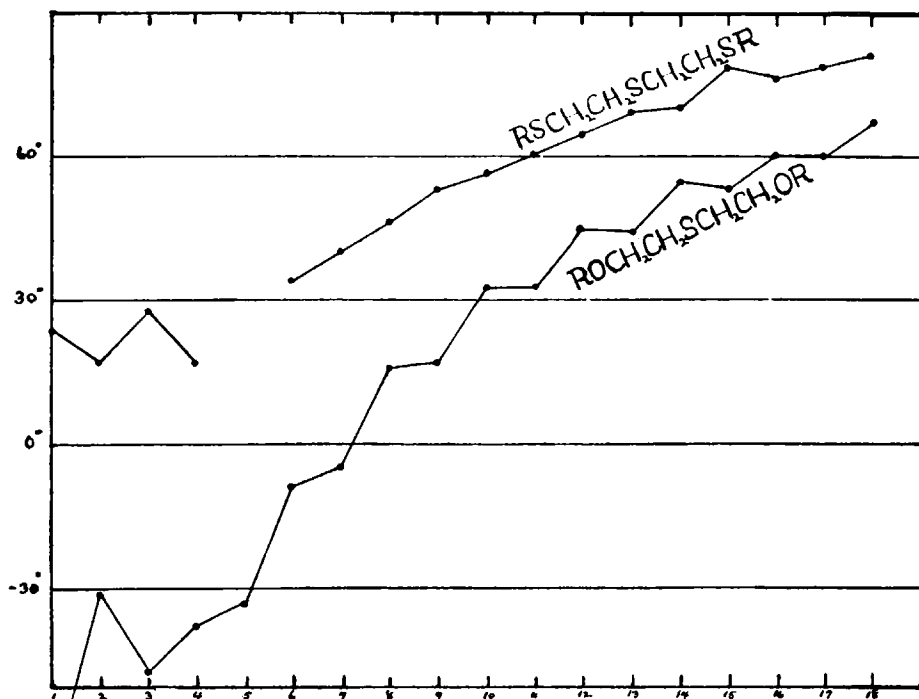


FIGURE 6.3. *Melting Points of tris-Sulfides, $RSCH_2CH_2SCH_2CH_2SR$, and of bis-Ether-Sulfides, $ROCH_2CH_2SCH_2CH_2OR$*

The melting points of a number of aliphatic sulfides are in Table 7.3. Inspection shows the surprising fact that the isomeric sulfides, on any one of the diagonals that slope upward to the right, melt at nearly the same temperature. Thus the sulfides of 23 carbon atoms, 19-4, 18-5, 17-6, etc., melt at 36.6°, 34.0°, 34.6°, 34.6°, 35.0°, 36.2°, 37.0°, and 37.2°. The melting points rise somewhat as the sulfur atom approaches the middle of the chain.

The data of Table 7.3 have been rearranged in Table 8.3 so as to place the melting points of isomeric sulfides on horizontal lines. The column at the extreme right gives the total number of carbon atoms. The melting points of the normal paraffins are in the next. The figures across the top are the numbers of carbon atoms in the smaller alkyl groups. Subtracting one of these from the number at the left gives the other alkyl radical.

TABLE 5.3

Melting Points (°C) of Some Sulfides and Ethers

R	Ph X R			<i>p</i> -MeC ₆ H ₄ X R			<i>p</i> -BrC ₆ H ₄ X R		
	S	Dif- ference	O	S	Dif- ference	O	S	Dif- ference	O
9	21.0	—	12.0	—	—	—	32.0	—	—
10	20.0	7.0	13.0	—	—	—	37.4	—	—
11	33.8	7.2	26.6	29.8	—0.6	30.4	42.0	—	—
12	33.0	8.0	25.0	31.0	5.4	25.6	46.8	12.4	34.4
13	43.8	7.6	36.2	40.2	—0.6	40.8	51.2	10.8	40.4
14	42.6	6.6	36.0	41.0	3.8	37.2	54.9	12.0	42.8
15	51.0	6.2	44.8	48.8	0.8	48.0	57.8	9.2	48.6
16	50.8	6.4	44.4	48.0	3.4	44.6	61.2	11.4	49.8
17	57.6	5.8	51.8	56.0	2.0	54.0	63.2	8.8	54.4
18	57.0	6.0	51.0	54.2	2.4	51.8	65.8	10.8	55.0
19	62.4	6.4	56.0	61.0	1.6	59.4	67.8	7.4	60.4
20	62.4	6.0	56.4	60.4	2.6	57.8	70.4	10.2	60.2

TABLE 6.3

Melting Points (°C) of Some Sulfides and Ethers

R	<i>p</i> -PhC ₆ H ₄ X R			β -C ₁₀ H ₇ X R			S(CH ₂ CH ₂ X R ₂)		
	S	Dif- ference	O	S	Dif- ference	O	S-S-S-	Differ- ence	O-S-O
1	73.0	—16.2	89.2	64.0	—8.0	72.0	24.3	101.3	—77.0
2	73.6	—1.8	75.4	16.0	—21.5	37.5	17.0	48.5	—31.5
3	72.5	—3.7	76.2	—	—	40.6	27.5	75.0	—47.5
4	71.5	—3.5	75.0	—	—	35.5	17.5	55.5	—38.0
5	76.7	10.7	66.0	9.0	—16.0	25.0	—	—	—33.0
6	76.5	10.7	65.8	19.0	8.0	11.0	34.0	53.0	—9.0
7	84.3	11.3	73.0	32.4	—2.6	35.0	40.0	45.0	—5.0
8	83.5	11.5	72.0	31.0	5.6	25.4	46.0	30.0	15.5
9	89.5	11.5	78.0	39.8	—6.8	46.6	53.0	36.0	17.0
10	88.0	11.0	77.0	42.0	4.0	38.0	56.0	23.7	32.3
11	93.0	10.0	83.0	46.8	—7.2	54.0	60.0	27.5	32.5
12	92.0	9.4	82.6	50.6	2.6	48.0	64.4	19.9	44.5
13	97.0	9.2	87.8	54.6	—6.6	61.2	68.8	28.8	44.0
14	96.2	10.2	86.0	59.0	3.8	55.2	70.0	16.0	54.0
15	99.0	9.0	90.0	61.0	—5.2	66.2	78.0	25.0	53.0
16	97.5	7.1	90.4	61.0	0.0	61.0	76.0	15.7	60.3
17	101.0	8.0	93.0	66.2	—4.8	71.0	78.2	18.2	60.0
18	100.5	8.3	92.2	66.0	0.6	65.4	80.6	14.0	66.5
19	—	—	—	71.2	—3.4	74.6	—	—	—
20	—	—	—	70.6	0.2	70.4	—	—	—

TABLE 7.3
Melting Points (°C) of Some Alkyl Sulfides, RSR'

R	R' = 1	2	3	4	5	6	7	8	9	10	11	12	13	14
10	—	—	—	—	—	—	—	—	20.0	27.0 ^a				
11	—	—	—	—	—	—	—	19.5	21.0	27.8	34.8			
12	—	—	—	—	—	10.0	18.0	21.0	31.0	32.8	37.2 ^b	42.2		
13	—	—	—	—	—	18.0	20.0	27.8	30.8	37.0	39.2	39.8	48.2	
14	—	—	—	—	18.5	19.0	28.2	30.0	36.2	37.8	44.2	46.0	49.2	53.8
15	—	—	—	18.0	18.0	28.2	29.2	35.0	37.2	42.6	44.6	49.2 ^b	—	50.6
16	—	19.0 ^c	—	20.8	—	29.2	34.6	36.6	42.6	43.6	49.0	50.0	—	55.6
17	26.2	26.2	—	28.2	31.4	34.6	36.2	41.0	43.2	47.4	48.0	53.6 ^b	—	57.4
18	—	32.1	—	32.0	34.0	36.4	41.4	42.6	47.6	48.0	53.0	53.8	—	59.4
19	36.0	36.2	36.0	36.6	37.0	41.8	42.8	47.2	48.6	51.8	—	53.2 ^b	—	61.6
20	41.0	41.6	—	41.0	41.0	43.0	47.4	48.4	52.0	52.8	57.0	57.8	—	61.6

The data are from the investigation ¹⁰⁸⁰ mentioned before, except as noted. As these were small scale preparations, the melting points below 25° are only approximate.

8 ^a Ref. 685; ^b Ref. 660; ^c Ref. 162.

TABLE 8.3

Melting Points (°C) of Isomeric Sulfides

No.	Hc. ^a	1	2	3	4	5	6	7	8	9	10	11	Av. ^b	Dep. ^c
18	28.0	26.2	19.0	—	—	—	10.0	—	—	—	—	—	10.0	18.0
19	31.4	—	26.2	—	18.0	18.5	18.0	18.0	19.5	20.0	—	—	18.6	12.8
20	36.6	36.0	32.1	—	20.8	18.0	19.0	20.0	21.0	21.0	27.0 *	—	19.9	16.7
21	40.3	41.0	36.2	—	28.2	—	28.2	28.2	27.8	(31.0)	27.8	—	28.1	12.2
22	44.4	—	41.6	36.0	32.0	31.4	29.2	29.2	30.0	30.8	32.8	34.8 *	30.8	13.6 *

No.	Hc. ^a	4	5	6	7	8	9	10	11	12	13	Av. ^b	Dep. ^c
23	47.3	36.6	34.0	34.6	34.6	35.0	36.2	37.0	37.2	—	—	35.5	11.8
24	50.7	41.0	37.0	36.4	36.2	36.6	37.2	37.8	39.2	42.2 *	—	37.2	13.5
25	53.2	—	41.0	41.8	41.4	41.0	42.6	42.6	(44.2)	39.8	—	41.5	11.7
26	56.4	—	—	43.0	42.8	42.6	43.2	43.6	44.6	46.0	48.2 *	43.7	12.7

No.	Hc. ^a	7	8	9	10	11	12	13	14	15	Av. ^b	Dep. ^c
27	58.5	47.4	47.2	47.6	47.4	49.0	49.2	49.2	—	—	48.1	10.4
28	62.1	—	48.4	48.6	48.0	—	50.0	—	53.8 *	—	48.7	12.5
29	63.3	—	—	52.0	51.8	53.0	53.6	—	50.6	—	52.2	11.1
30	65.8	—	—	—	52.8	—	53.8	—	55.6	59.0 *	54.0	11.8
31	67.2	—	—	—	—	57.0	—	—	57.4	—	57.2	10.0
Average												12.8

* The symmetrical sulfides which melt about 5° higher than the average of their unsymmetrical isomers and are not included in the averages. The averages are taken to the right of the vertical lines. Two melting points, which seem out of line, are excluded.

^a Hydrocarbon; ^b Average; ^c Depression.

The melting points of the methyl alkyl sulfides, 1-17, 1-19, and 1-20 are close to those of the hydrocarbons of the same number of carbon atoms. The ethyl sulfides melt lower and the propyl and butyl still lower. Starting with the butyl sulfides, the melting points of any one group of isomers are close together, until the sulfur atom approaches the center of the chain, when they rise. The data are plotted in Figure 7.3. Averages have been taken of the melting points over the nearly constant range and are given in the column marked *Av.* The depression, which is in the

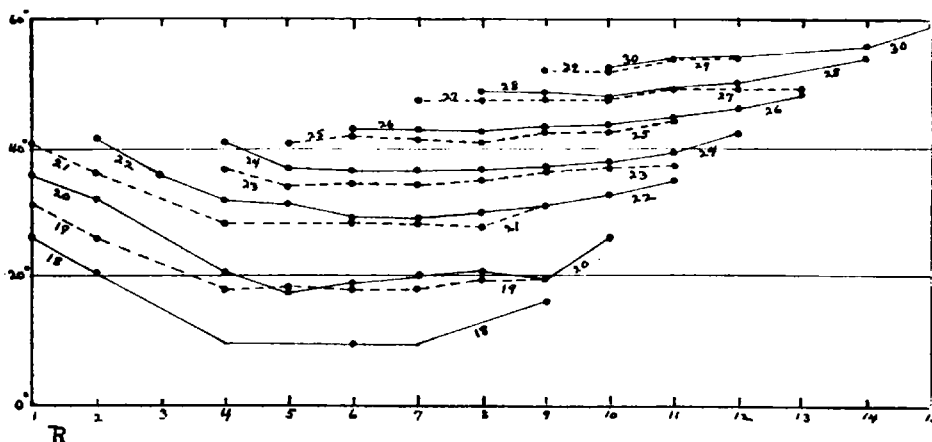


FIGURE 7.3. *Melting Points of Isomeric Sulfides, RSR'*

The figures on the lines designate the total number of carbon atoms and R is the smaller alkyl.

last column, is the difference between this average and the melting point of the hydrocarbon. It will be noted that these depressions are consistently lower for the odd groups than for the even. The averages show regular alternation. This is evident in Figure 7.3. Thus the broken lines which join the melting points of the odd sulfides are much nearer to the solid lines above them, which join the even sulfides, than to the solid lines below. These averages are plotted in Figure 8.3 along with the melting points of the hydrocarbons.

The melting points of a number of *bis*-sulfides, $\text{RS}(\text{CH}_2)_n\text{SR}$, are given in Table 9.3.

In Figure 9.3, these are plotted against the number of carbon atoms in R, the value of n being marked on each curve. The melting-point pattern for the sulfides in which $n = 2$ is strikingly unlike any of the others. The melting points are relatively high and there is pronounced alternation. For other values of n there

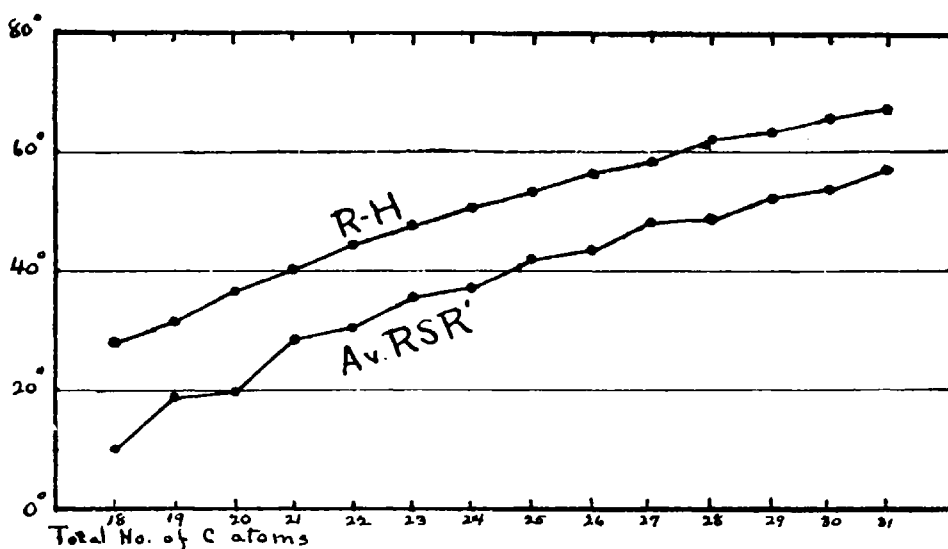


FIGURE 8.3. Averages of the Melting Points of the Isomeric Sulfides Compared with the Melting Points of the Hydrocarbons of the Same Number of Carbon Atoms

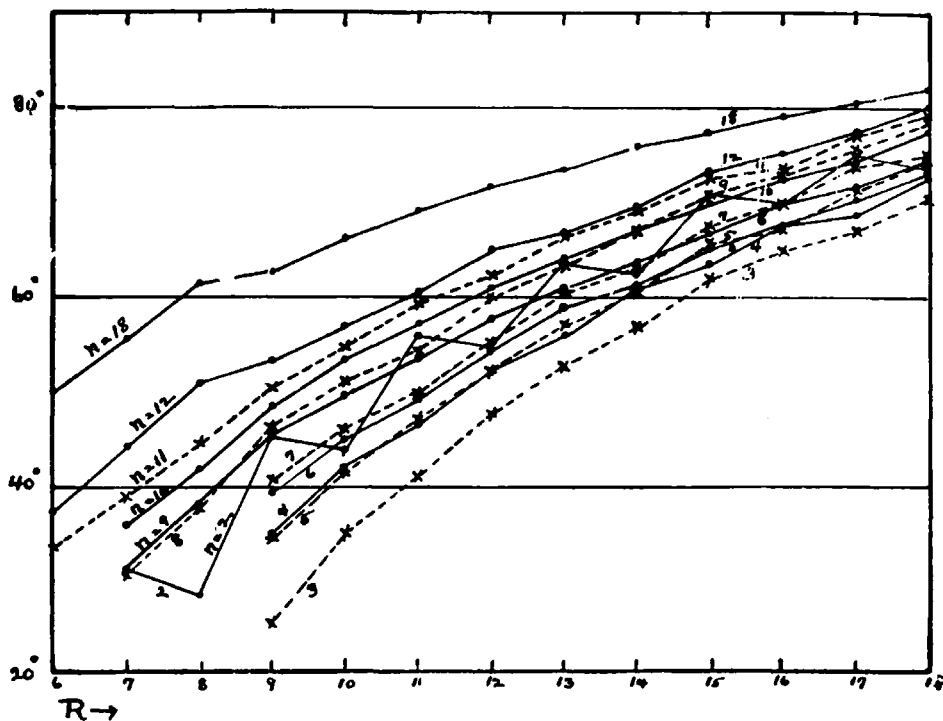


FIGURE 9.3. Melting Points of the bis-Sulfides, $RS(CH_2)_nSR$, Plotted against R

TABLE 9.3

Melting Points (°C) of RS(CH₂)_nSR

R	n = 1	2	3	4	5	6	7	8	9	10	11	12	18
6	—	—	—	—	—	—	—	—	—	—	33.6	37.6	52.6
7	—	31.2	—	—	—	—	—	31.6	31.0	36.2	39.2	44.6	56.0
8	—	29.0	—	—	—	—	—	38.8	38.2	42.2	45.2	51.6	61.6
9	—	45.2	26.5	35.0	34.4	39.4	40.8	44.6	46.2	48.6	50.4	53.2	62.8
10	—	44.0	35.2	42.2	41.2	45.0	46.2	49.6	51.0	53.4	54.8	57.0	66.0
11	—	(53.6)	41.2	46.8	47.2	49.4	50.0	51.2	54.4	57.2	59.6	60.6	69.2
12	42.5	55.0	47.8	52.2	52.2	54.8	55.4	57.8	60.0	61.0	62.4	65.0	71.6
13	—	64.0	53.0	56.2	57.4	59.2	60.4	61.2	63.8	64.2	66.6	67.0	73.6
14	50.0	63.0	57.0	61.2	61.0	62.0	63.4	64.0	67.2	67.2	69.2	69.8	76.0
15	—	70.8	62.0	63.4	65.6	65.4	67.6	66.8	70.8	70.0	72.4	73.4	77.2
16	59.0	70.0	65.0	67.8	67.4	68.0	70.0	70.0	72.8	72.4	73.8	75.2	79.2
17	—	75.0	67.2	69.0	71.6	70.6	73.6	72.0	75.8	74.8	77.0	77.6	80.2
18	—	73.6	71.0	72.8	74.0	73.4	73.0	74.0	78.0	77.4	78.6	79.4	83.0

is little alternation. When $n = 4, 6,$ or 8 , the melting points are close to those of the next higher series, in which $n = 5, 7,$ or 9 . This shows up more plainly in Figure 10.3 in which the same data are plotted against n .

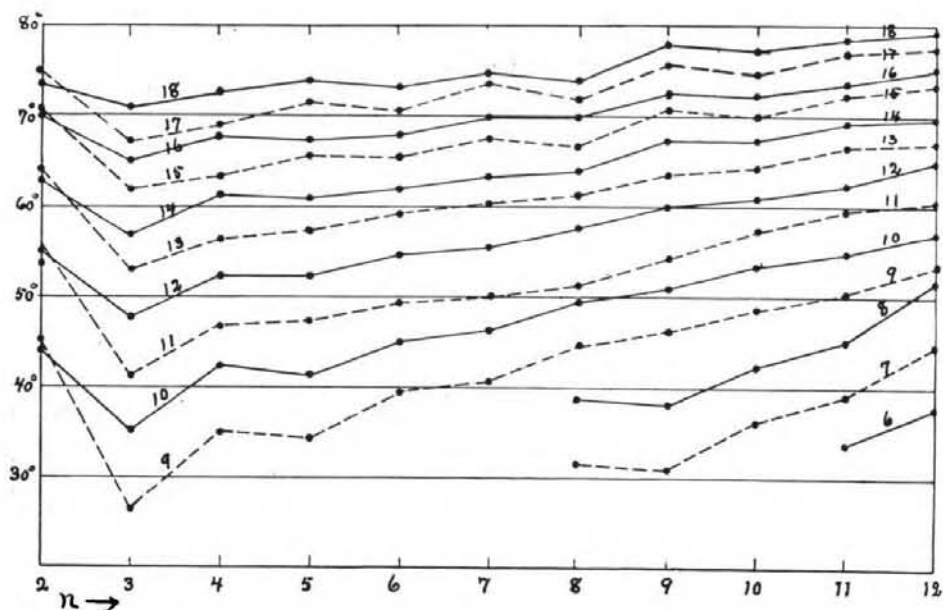


FIGURE 10.3. *Melting Points of the bis-Sulfides, $RS(CH_2)_nSR$, Plotted against n*

In Figure 10.3 it will be noted that when $n = 2$ the sulfides, having odd-numbered alkyl groups, melt above the next higher even. There is in each case a sharp drop from $n = 2$ to $n = 3$ from which there is a rise to $n = 4$. There is alternation in all of the series. This dies down as n increases. With the higher alkyl radicals, there is a reversal of phase. With undecyl this occurs at $n = 9$, with tridecyl, myristyl, and cetyl at 7, and with pentadecyl, heptadecyl, and octadecyl at 5. This means that up to these values of n , the sulfides in which n is an odd number melt below a smooth curve and above this curve for higher values of n .

In Figure 11.3, the melting points are plotted against the total number of carbon atoms, for the normal hydrocarbons, the bis-sulfides $RS(CH_2)_2SR$, the tris-sulfides, $RS(CH_2)_2S(CH_2)_2SR$ and the averages of RSR' from Table 8.3 and $RS(CH_2)_nSR$ from Table 11.3. It will be noted that the even-numbered bis-sulfides melt close to the tris-sulfides and above the hydrocarbons. The averages of the melting points of RSR' and $RS(CH_2)_nSR$ are

TABLE 10.3

Melting Points (°C) of RS(CH₂)_nSR, n Even

No.	Hydro-carbon	n = 2	4	6	8	10	12	18	Average	Depression
22	44.4	44.0	35.0	—	31.6	—	—	42.2 ^a	31.6	12.8
24	50.7	53.6	42.2	39.4	38.8	36.2	37.6	41.2 ^a	38.0	12.7
26	56.4	55.0	46.8	45.0	44.6	42.2	44.6	44.8	44.2	12.2
28	61.2	64.0	52.2	49.4	49.6	48.6	51.6	47.2	49.3	11.9
30	65.8	63.0	56.2	54.8	51.2	53.4	53.2	52.6	53.0	12.8
32	69.5	70.8	61.2	59.2	57.8	57.2	57.0	56.0	57.4	12.1
34	72.6	70.0	63.4	62.0	61.2	61.0	60.6	61.6	61.3	11.3
36	75.8	75.0	67.8	65.4	64.0	64.2	65.0	62.8	64.3	11.5
38	79.3	73.6	69.0	68.0	66.8	67.2	67.0	66.0	67.0	12.3
40	81.8	—	72.8	70.6	70.0	70.0	69.8	69.2	69.9	11.9
42	84.2	—	—	73.4	72.0	72.4	73.4	71.6	72.6	11.6
44	86.5	—	—	—	74.0	74.8	75.2	73.6	74.4	12.1
46	88.6	—	—	—	—	77.4	77.6	76.0	77.0	11.6
48	90.6	—	—	—	—	—	79.4	77.2	78.3	12.3
50	92.4	—	—	—	—	—	—	79.2	—	13.2 ^b
52	94.1	—	—	—	—	—	—	80.2	—	13.9 ^b
54	95.6	—	—	—	—	—	—	83.0	—	12.6 ^b
Average Depression		—	9.1	11.0	12.1	12.3	11.6	12.9		12.1

The averages are taken of the data to the right of the vertical line. As is shown in Figure 9.3, when $n = 2$ the melting points show decided alternation and form a pattern which is quite different from the others. When $n = 4$ the melting points are about 2° higher than those of the other isomers.

^a These melting points are high on account of the proximity of the sulfur atoms to the ends of the chains, R being ethyl in one case and propyl in the other.

^b These are excluded as they are not from averages.

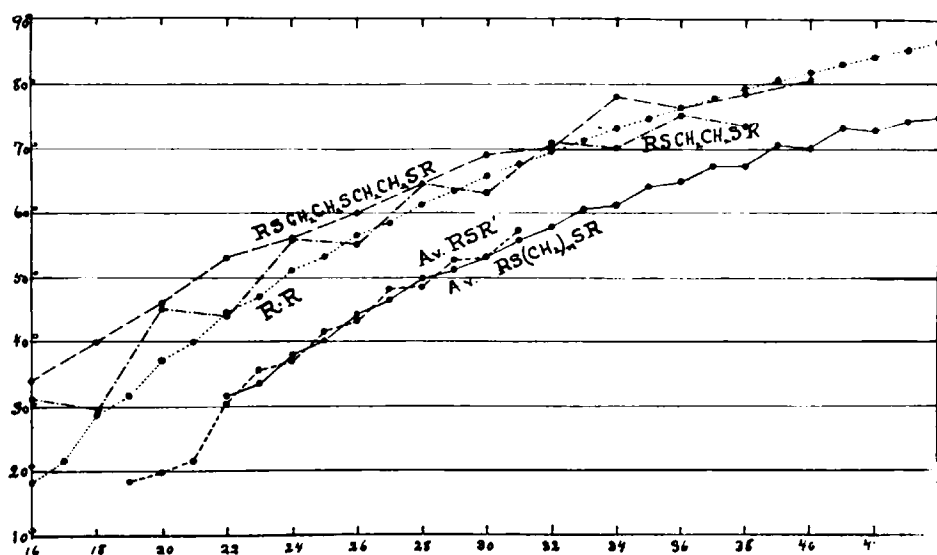


FIGURE 11.3. Melting Points of the bis-Sulfides, $RSCH_2CH_2SR$, the tris-Sulfides, $RSCH_2CH_2SCH_2CH_2SR$, and the Hydrocarbons, RR , Compared with the Average of the Melting Points of the Isomeric Sulfides, RSR' , and the Isomeric bis-Sulfides, $RS(CH_2)_nSR$, Plotted against the Total Number of Carbon Atoms

close together and show regular alternation. They are below the melting points of the hydrocarbons.

TABLE 11.3

Melting Points ($^{\circ}C$) of $RS(CH_2)_nSR$, n Odd

No.	Hydro-carbon	1	3	5	7	9	11	Aver-age	Depres-sion
23	47.3	—	35.2	34.4	—	31.0	33.6	33.6	13.7
25	53.2	42.5	41.2	41.2	40.8	38.2	39.2	40.5	12.7
27	58.5	—	47.8	47.2	46.2	46.2	45.2	46.5	12.0
29	63.3	50.0	53.0	52.2	50.0	51.0	50.4	51.1	12.2
31	67.2	—	57.0	57.4	55.4	54.4	54.8	55.8	11.4
33	71.6	59.0	62.0	61.0	60.4	60.0	59.6	60.3	11.3
35	74.4	—	65.0	65.6	63.4	63.8	64.4	64.4	10.0
37	77.6	—	67.2	67.4	67.6	67.2	66.6	67.2	10.4
39	80.1	—	71.0	71.6	70.0	70.8	69.2	70.5	9.6
41	83.0	—	—	74.0	73.6	72.8	72.4	73.2	9.8
43	85.3	—	—	—	73.0	75.8	73.8	74.2	11.1
45	87.5	—	—	—	—	78.0	77.0	77.5	10.0
47	89.6	—	—	—	—	—	78.6	78.6	11.0
Average Depression			10.4	10.4	11.4	11.7	11.8	Av. 11.1	11.2

Physical Properties of Sulfides

GENERAL

In the following tables, data on the physical properties of sulfides are assembled. No claim is made for completeness.

The ultraviolet absorption of sulfides has been studied by many.^{58, 86, 177, 186, 286.7, 441, 483, 650, 698, 774, 838, 899, 903b, 1031.5, 1035, 1045, 1050, 1221, 1257, 1284a, 1285} Recently a comprehensive investigation has appeared.^{412a} The infrared absorption has been determined for a number of alkyl sulfides.^{92, 395, 433, 753b, 967, 1024, 1081.5, 1050, 1156, 1176, 1284b, 1303} The vibration frequency assigned to the stretching of the bonds C—S—C in aliphatic sulfides is 14.41 to 16.00.¹¹⁷² The bond energy for C=S is 117.8, for C—S 59.2, and for S—H 87.1.¹²⁵³ The relation of bond constants to electronegativities of the atoms has been studied.⁵⁰⁴ The appearance potentials have been determined for the gaseous ions formed by electron impact on methyl and ethyl sulfides.⁴⁴³ The electric moments of several sulfides have been measured.⁸¹¹ Valence-force potentials have been measured for methyl sulfide and selenide.¹¹⁸⁷ Numerous measurements have been made on the Raman spectra of sulfides.^{150, 151, 370, 433, 502, 717, 864, 880, 890, 967, 1186, 1193, 1279, 1320, 1327}

There have been many determinations of the dipole moments of sulfides, particularly for the purpose of finding the valence angle of the sulfur atom.^{98, 99, 102, 536, 537, 623a, 761, 805, 806, 903b, 967, 1113.5, 1246, 1247, 1301a, 1340, 1341, 1384} The molecular refractivities of sulfur compounds have been measured in order to determine the atomic refractivity of sulfur in its various combinations.^{46b, 167, 332, 390, 863b, 863c, 935, 937a, 1034, 1325, 1326} The dispersion has also been considered.⁴⁹⁹ The influence of sulfur in optical rotation has been evaluated.^{206b, 1185, 1399} The relation between internuclear distances and potential barriers has been studied.⁴⁴⁴ Some phenyl sulfides have been studied polarographically.^{317.5}

Vapor pressures,^{83, 108, 1074a, 1074b} critical temperatures,^{108, 419, 574} surface tensions,^{78, 108, 903a, 1324.5, 1326, 1336a} and parachors^{78, 332, 481, 903a, 1324.5, 1325, 1326} have been measured. The vapor pressure-temperature relationships have been determined for several sulfides and other sulfur compounds.¹³⁶⁵ Equations have been derived connecting vapor pressure with temperature and heats of vaporization with pressure and temperature.⁵²⁹ The heats of

formation of sulfides have been compared with those of mercaptans and of carbon disulfide.¹²⁵⁰ The heat capacity, 13.3 to 292°K, the heat of fusion, vapor pressure, 250.6 to 290.2°K,⁹⁵⁷ electron diffraction,²⁰⁷ internal rotation,³⁷ and the adsorption on palladium³⁶⁴ have been determined for methyl sulfide; the heat of vaporization,^{957, 966, 1286} entropy,^{957, 966} ionization potential,¹²⁴² dielectric constants,^{623a, 741, 1336a, 1340} and association have been measured for methyl and ethyl sulfide,^{1336b} the magnetic susceptibility has been determined for these and propyl,^{974a} the association,²⁶⁴ heat conductivity, and specific heat¹³⁴⁴ for ethyl and the viscosities for methyl,^{81, 1290} ethyl,^{81, 109, 1290} butyl, and *i*-octyl.¹⁰⁹ The diamagnetic susceptibilities are: 44.91 for methyl, 67.88 for ethyl, and 91.82 for propyl³¹⁴ sulfides. The specific magnetic rotation of phenyl sulfide is 3.211 at 15°.⁹⁸⁷ Entropy, free energy, and heat capacities of methyl^{79, 117} and ethyl⁷⁹ sulfides have been determined over a wide temperature range. The entropy of vaporization of methyl sulfide has been calculated.¹²²⁰ The heat of combustion for methyl sulfide is 455.6 kilocalories per mole and that for ethyl is 769.2.⁵¹⁰ The densities and viscosities have been determined for a number of alkyl phenyl sulfides at 22°^{1324.5} and for nonyl and cetyl phenyl sulfides at intervals from 20 to 300°.¹⁴⁴ Complete thermodynamic investigations have been made of methyl-ethyl¹¹⁶² and of diethyl¹¹⁶¹ sulfides.

Diamagnetic properties of phenyl sulfide and selenide have been determined.^{974d} The halochromy of nitrothiophenyl ethers has been investigated.⁹⁹⁶ The contact angle for water and benzyl sulfide has been measured by the plate method.⁹⁴⁵

The crystal structures of compounds of the general formula $C(SR)_4$,^{55c} particularly the tetramethyl, $C(SMe)_4$, which has transition points at 23.2 and 45.5°, have been studied.^{54, 985, 986a, 986b} Crystallographic data for benzyl sulfide^{436, 734} and other aromatic^{1301b} sulfides have been presented. In the decamethylene ether of 4,4-dihydroxydiphenyl sulfide, the distance of the sulfur from the rings is 1.71Å and the angle between the two sulfur valencies 112.4°.⁷¹⁶ The formation of mixed crystals of a number of aromatic sulfides has been investigated.^{1092, 1093, 1094} Phenyl sulfide, selenide, and telluride are isomorphous.^{974b} The structures of MeSOMe and MeCOMe have been compared; the sulfoxide is pyramidal while acetone is planar.⁸⁰

AZEOTROPES

A large number of azeotropes, involving the lower alkyl sulfides, have been included in two comprehensive treatises⁷⁵¹ and various articles⁷⁵² by Lecat, from which the majority of the data of Table 12.3 are taken. Since sulfides of this class are found in petroleum distillates, azeotropes with hydrocarbons have been investigated by the petroleum chemists, Desty and Fidler.³⁶¹ Azeotropes with perfluor compounds have been recommended for separating sulfides from hydrocarbons.³⁰¹

TABLE 12.3

*Azeotropes Involving Sulfides*Methyl Sulfide, Me_2S , b. 37.3°

Other component	Azeotrope			R_2S %	Reference
	B.p., $^\circ\text{C}$	B.p., $^\circ\text{C}$	Differ- ence		
Cyclopentane	49.3°	37.1°	0.2°	89	361
2-Methylbutene-1	31.2°	30.6°	0.6°	19	361
Ethyl ether	34.6°	34.0°	0.6°	20	752b
2,2-Dimethyl- butane	49.7°	36.5°	0.8°	84	361
Isopentane	27.9°	26.6°	1.3°	30	361
"	27.9°	27.3°	0.6°	15	608
Isoprene	34.3°	32.5°	1.8°	35	752b
Pentane	36.1°	31.8°	4.3°	50	361
"	36.1°	33.9°	2.2°	47	752b
"	36.2°	33.5°	2.7°	45	608
2-Methylbutene-2	38.6°	34.8°	2.5°	57	361
"	37.2°	34°	3.2°	52	751a, b
Methyl formate	31.7°	29.0°	2.7°	38	752b
Methanol	64.7°	34°	3.3°	85	751a

Methyl Ethyl Sulfide, MeSEt , b. 66.6°

2,2-Dimethyl- pentane	79.2°	66.4°	0.2°	91	361
2,3-Dimethyl- butane	58.1°	57.4°	0.7°	21	361
Hexene-1	63.5°	62.7°	0.8°	36	361

TABLE 12.3 (Continued)
Azeotropes Involving Sulfides

Other component	Azeotrope		Difference	R ₂ S %	Reference
	B.p., °C	B.p., °C			
Methylcyclopentane	71.8°	65.6°	1.0°	64	361
Hexane	68.7°	63.9°	2.7°	57	361
Ethyl Sulfide, Et ₂ S, b. 92.1°					
2,4-Dimethylpentane	80.5°	80.5°	0.0°	2	361
Methylcyclohexane	101.0°	92.1°	0.0°	95	361
Acetic acid	118.1°	91.5°	0.6°	90	752b
2,2,4-Trimethylpentane	99.3°	91.4°	0.7°	81	361
1,1-Dimethylcyclopentane	87.9°	87.0°	0.9°	28	361
Methyl isopropyl ketone	95.4°	90.5°	1.6°	70	752b
1,2-Diethoxyethane	87.9°	85.9°	2.0°	35	752b
2,3-Dimethylpentane	89.9°	87.9°	2.0°	41	361
Methyl ethyl ketone	79.6°	77.5°	2.1°	20	752a
3-Methylhexane	91.6°	89.2°	2.4°	51	361
<i>t</i> -Butanol	82.4°	79.8°	2.6°	30	752b
“	82.6°	78.2°	4.4°	50	751a
Ethyl nitrate	87.7°	85.0°	2.7°	42	752b
Methyl carbonate	90.2°	86.8°	3.4°	53	752b
Methanol	64.7°	61.2°	3.5°	38	752b
Ethyl acetate	77.1°	73°	4.1°	77	752a
Ethanol	78.3°	72.6°	5.7°	44	751a
Allyl alcohol	96.8°	85.0°	7.1°	70	752b
Nitromethane	101.2°	85.0°	7.1°	70	752b
Propanol	97.2°	85.0°	7.1°	70	751b
Formic acid	100.7°	82.2°	9.9°	65	752b

TABLE 12.3 (Continued)
Azeotropes Involving Sulfides

Other component	Azeotrope			R ₂ S %	Reference
	B.p., °C	B.p., °C	Differ- ence		
Dichlorobromo- methane *	90.1°	96.7°	4.6°	42	751a
Methyl Propyl Sulfide, MeSP _r , b. 95.5°					
Ethylcyclo- pentane	103.4°	95.4°	0.1°	91	361
2,2-Dimethyl- hexane	106.8°	95.4°	0.1°	95	361
1,1-Dimethyl- cyclopentane	87.9°	87.7°	0.2°	10	361
Methylcyclo- hexane	101.0°	95.1°	0.4°	80	361
2,3-Dimethyl- pentane	89.9°	89.1°	0.8°	25	361
3-Methylhexane	91.6°	90.5°	1.1°	35	361
2,2,4-Trimethyl- pentane	99.3°	94.0°	1.5°	68	361
Methyl <i>i</i> -Propyl Sulfide, MeSCHMe ₂ , b. 84.8°					
3-Methylhexane	91.6°	84.4°	0.4°	84	361
2,2-Dimethyl- pentane	79.2°	78.4°	0.8°	25	361
2,3-Dimethyl- pentane	89.9°	83.9°	0.9°	75	361
Cyclohexane	80.8°	79.8°	1.0°	29	361
2,4-Dimethyl- pentane	80.5°	79.4°	1.1°	32	361
1,1-Dimethyl- cyclopentane	87.9°	83.6°	1.2°	67	361
Propyl Sulfide, Pr ₂ S, b. 141.5°					
Ethyl isovalerate	134.7°	134.0°	0.7°	90	751a
2-Methylpyridine	130.7°	129.8°	0.9°	10	752b

* This azeotrope boils above either of the components.

TABLE 12.3 (Continued)
Azeotropes Involving Sulfides

Other component	Azeotrope		Difference	R ₂ S %	Reference
	B.p., °C	B.p., °C			
Acetic acid	118.1°	116.9°	1.2°	27	752b
Butyl ether	142.4°	140.3°	1.2°	62	752b
Butanol	117.6°	116.0°	1.6°	25	751a
Pyrrole	130.0°	127.5°	2.5°	35	752b
Formic acid	100.7°	98.0°	2.7°	27	752b
2-Chloroethanol	128.6°	125.5°	3.1°	33	752b
<i>i</i> -Amyl alcohol	131.3°	127.5°	3.8°	40	751a
Propionic acid	141.3°	136.5°	4.8°	55	752b
Cellosolve	135.3°	130.2°	5.1°	48	752b
Bromoform *	149.5°	151.5°	2.0°	10	752b
<i>i</i> -Propyl Sulfide, Me ₂ CHSCHMe ₂ , b. 120.5°					
Methyl <i>i</i> -butyl ketone	116.1°	114.9°	1.2°	28	752b
Pyrazine	117.2°	116.0°	1.2°	25	752b
Methyl butyl ketone	123.3°	119.0°	1.5°	68	752b
<i>i</i> -Butyl acetate	117.4°	115.2°	2.2°	43	752b
<i>i</i> -Butyl alcohol	108.0°	105.8°	2.2°	27	752b
Pyrrole	130.0°	117.5°	3.0°	80	752b
Epichlorohydrin	116.4°	111.5°	4.9°	33	752b
2-Chloroethanol	128.6°	115.5°	5.0°	70	752b
Butanol	117.8°	112.0°	5.8°	55	752b
Acetic acid	118.1°	111.5°	6.6°	52	752b
Formic acid	100.7°	93.5°	7.2°	93	752b
Butyl Sulfide, Bu ₂ S, b. 185.0°					
β,β'-Dichloroethyl ether	178.6°	178.4°	0.2°	12	752b
Butylbenzene	183.1°	182.0°	1.1°	40	752b
<i>o</i> -Cresol	191.1°	183.8°	1.2°	75	752b
<i>i</i> -Valeric acid	176.5°	175.0°	1.5°	27	752b
<i>o</i> -Chlorophenol	176.8°	175.0°	1.8°	18	752b
Phenol	182.2°	177.5°	4.7°	55	752b

* This azeotrope boils higher than either constituent.

TABLE 12.3 (Continued)
Azeotropes Involving Sulfides

Other component	Azeotrope		Differ- ence	R ₂ S %	Reference
	B.p., °C	B.p., °C			
Acetamide	222.1°	180.0°	5.0°	92	752b
Methyl malonate	181.4°	176.2°	5.2°	50	752b
<i>i</i> -Butyl Sulfide, <i>i</i> -Bu ₂ S, b. 172.0°					
Ethyl aceto- acetate	180.4°	171.0°	1.0°	90	752b
<i>i</i> -Amyl ether	173.2°	171.0°	1.0°	62	752b
Methyl hexyl ketone	172.8°	169.8°	2.2°	50	752b
<i>o</i> -Chlorophenol	176.8°	169.5°	2.5°	72	752b
Methyl lactate	171.7°	169.0°	2.7°	52	752b
Methyl aceto- acetate	169.5°	166.0°	3.5°	42	752b
Ethyl carbamate	185.2°	166.5°	5.5°	77	752b
Butyl Cellosolve	171.1°	163.8°	7.3°	58	752b
Ethanolamine	170.8°	156.0°	14.8°	67	752b
<i>i</i> -Amyl Sulfide, <i>i</i> -Am ₂ S, b. 214.8°					
1,3,5-Triethyl- benzene	215.5°	214.0°	0.8°	65	752b
Methyl α -ter- penyl ether	216.2°	213.8°	1.0°	70	752b
Methyl maleate	204.0°	203.0°	1.0°	18	752b
<i>p</i> -Chlorophenol	219.7°	212.5°	2.3°	72	752b
<i>o</i> -Nitrophenol	217.2°	212.5°	2.3°	70	752b
Propionamide	222.2°	204.0°	10.8°	80	752b
Acetamide	222.1°	199.5°	15.3°	83	752b
Methyl Allyl Sulfide, MeSCH ₂ CH:CH ₂ , b. 92.2°					
Methanol	64.5°	61.8°	2.7°	40	1031
Allyl Sulfide, (CH ₂ :CHCH ₂) ₂ S, b. 139.3°					
Ethyl carbonate	126.5°	126.0°	0.5°	10	752b
Ethyl chloroacetate	143.5°	138.5°	0.8°	78	752b

TABLE 12.3 (Continued)
Azeotropes Involving Sulfides

Other component	Azeotrope			R ₂ S %	Reference
	B.p., °C	B.p., °C	Differ- ence		
Dipropyl ketone	143.5°	138.2°	1.1°	75	752b
Acetic acid	118.1°	116.5°	1.6°	21	752b
Methyl Cello- solve	124.5°	122.5°	2.0°	25	752b
Ethanolamine	170.8°	137.2°	2.1°	92	752b
Ethyl butyrate	119.9°	117.5°	2.4°	15	752a
Mesityl oxide	130.5°	128°	2.5°	25	752a
Propionic acid	141.3°	136.5°	2.8°	60	751c, 752b
Pyrrole	130.0°	127.0°	3.0°	30	752b
Formic acid	100.7°	97.5°	3.2°	20	752b
<i>i</i> -Amyl formate	123.6°	120°	3.6°	20	752b
β-Picoline	144.0°	135.5°	3.8°	70	752b
β-Bromoethanol	150.2°	135.5°	3.8°	80	752b
β-Chloroethanol	128.6°	124.5°	4.1°	39	751c, 752b
Epibromohydrin	138.5°	133.3°	5.2°	40	752b

In each group, the azeotropes are arranged in the order of increasing difference between the boiling point of the azeotrope and that of the lower of the two components. It will be noted that these differences are quite small. About one fourth of the azeotropes listed boil 1° or less below the lower of the two components. In about the same number, these differences exceed 3°. The alkyl sulfides are only slightly associated and so are the hydrocarbons. Large depressions are not to be expected. Pronounced azeotropes are formed with alcohols, acids, and amides. In two cases the azeotropes boil higher than either of the components.

LISTS OF PHYSICAL PROPERTIES OF SULFIDES

The physical properties of a large number of sulfides are given in the following tables. Satisfactory data are available for some, but are sadly lacking for the majority. The remarks made in Chapter 1, Volume I, concerning completeness and accuracy of data apply here also.

Where the boiling points found in the literature are given at a pressure near 760 mm., corrections have been made only where they seemed justified. If the boiling point is 158° at 754 mm., the correction would be about 0.3° , but to set down 158.3° would be misleading since it would indicate that the original reading had been made to 0.1° which was not the case.

Densities, such as 25/25, have been changed to 25/4 from the density of water at 25° .

The compounds have been grouped and an effort has been made to arrange those in each group in a logical order, but many have had to be thrown in. Naming has been abandoned for the more complicated compounds on account of the inordinate amount of space required.

Symmetrical Alkyl Sulfides

Methyl, Me_2S , m. -98.25° ,⁸¹⁹ -98.30° ,⁹⁵⁷ -83.2° ,^{108, 263, 1321} b. 37.65° ,⁵⁷⁴ 36.2° ,^{108, 1105, 1367} 37.5° ,^{353, 427, 1289, 1326} 37.52° ,¹²⁹⁰ 37.7° ,⁸⁷ 37.3° ,^{700d, 751a, 819} 37.2° ,^{359, 608, 1234} 37° ,^{353, 560.5, 1286, 1336b} 36° ,¹⁰⁸ 38° ,^{140, 263, 326, 427, 623b, 694, 1321, 1393} b_{760} $37.8-8.2^\circ$,¹³⁹⁴ 38.2° ,⁴¹⁹ 38.4° ,^{623b} b_{10} -49.2° , b_{20} -39.7° , b_{40} -28.4° , b_{100} -12.0° , b_{200} 2.6° , b_{400} 18.7° ,¹⁰⁸ b_{172} 0° , b_{296} 11.6° , b_{436} 20.8° , b_{727} 35.2° , b_{1075} 44.7° , b_{1572} 59.1° ; ¹⁰⁸ d 0/4 0.8702,^{1289, 1290} d 20/4 0.8476,^{623b} 0.8483,⁸¹⁹ 0.8449,¹²³⁴ 0.8456,¹³⁹⁴ d_{20} 0.845,¹⁰⁷⁷ 0.846,³⁵³ 0.854,¹³⁶⁷ d 21/4 0.8458,^{263, 1321} d 25/4 0.8424,⁸¹⁹ d at b.p. 0.82567;¹²⁸⁹ n 20/D 1.4356,^{623b} 1.4353,⁸¹⁹ 1.4578,¹³⁹⁴ 1.4349,^{560.5} n 25/D 1.4319;⁸¹⁹ critical temperature 229.8° ,¹⁰⁸ 231.19° ,⁵⁷⁴ 231.2° ; ^{419, 1321} critical pressure 56.14;¹³²¹ molar heat of vaporization 6910 cal.,¹²⁸⁶ 6688;⁹⁵⁷ heat of fusion 1908;⁹⁵⁷ entropy at 25° 46.94 cal/degree/mole, at 291.06°K and 365.5 mm., 69.35° cal/degree/mole;⁹⁵⁷ specific volume 75.1;¹²⁸⁹ dielectric constant at 20° , 6.2;^{1336a} heat of combination at constant pressure 457.35 cal.;¹²⁸⁷ magnetic susceptibility 468×10^{-7} .^{974a} See Table 1.3.

Ethyl, Et_2S , m. -103.91° ,⁸¹⁹ -103.93° ,^{529.5} -103.95° ,¹¹⁶¹ -102.5° ,^{1291a} -99.5° ,^{263, 1321} -103.3° ; ¹²⁹² b. 92° ,^{261a, 263, 353, 419, 694, 819, 1321} 92.1° ,^{87, 529.5, 919, 1161, 1290, 1292} 92.2° ,^{264, 586, 751a, 935} 92.3° ,^{359, 586, 608} 93° ,^{1336b} 92.6° ,⁵⁷⁴ 91.9° ,^{700d, 1234} 91.6° ,^{108, 1105, 1367} 91.5° ,^{623a, 1336c, 1336d} $93-4^\circ$,¹⁰⁹ 91.4° ,¹²⁸⁶ 91.2° ,^{46a} 91° ,^{253, 412c, 477b, 609, 740, 1001b} 90° ,^{57.5, 556.5} 90.4° ,^{903a} $90-2^\circ$,¹³⁹ b_{749} $90.5-1.5^\circ$,²²³ b_{10} 0.40° ,¹²⁹² b_{29} 10° , b_{48} 20° , b_{77} 30° ,⁸³ b_{106} 33.3° , b_{160} 43.3° , b_{502} 77.3° , b_{938} 98.8° ; ¹⁰⁸ d 0/4 0.8524,^{46a, 46b} 0.8563,¹⁰⁸

- 0.8565,^{1001b} d 0/0 0.83672,¹²⁹⁰ d 15/4 0.84102,¹²⁹² d₂₀ 0.837,³⁵³ 0.825,¹⁰⁷⁷ 0.836,¹³⁶⁷ d 20/4 0.8363,⁸¹⁹ 0.83623,^{529.5} 0.83676,³⁹⁰,⁹³⁵ 0.8331,^{46a, 46b} 0.83679,^{633a} 0.8367,^{76, 223, 919} 0.8278,¹⁰⁹ d 20.5/4 0.8362,²²³ d 21/4 0.8364,^{263, 1321} d 25/4 0.8316,⁹¹⁹ 0.8313,⁸¹⁹ 0.83120,^{529.5} 0.8299,^{46a, 46b, d₂₅} 0.825,²⁶⁴ d 29.1/4 0.8279,¹⁰⁸ d 30/4 0.82625,^{529.5} d₃₁ 0.8203,²⁶⁴ d 38.5/4 0.8183,¹⁰⁸ d 41.4/4 0.8155,¹³²⁶ d 49.8/4 0.8065, d 59.9/4 0.7952, d 79.6/4 0.7756, d 98.7/4 0.7552;¹⁰⁸ n 15/D 1.44550,¹²⁹² n 20/D 1.4425,⁹¹⁹ 1.4427,⁸¹⁹ 1.44298,^{529.5} 1.4428,^{46a, 46b} 1.44303,¹⁰⁹ 1.44233,^{623a, 935} n 20.5/D 1.44253,²²³ 1.4423,³⁹⁰ n 25/D 1.4395,^{46a, 46b} 1.4400,⁸¹⁹ 1.44017, n 30/D 1.43734;^{529.5} critical temperature 284.67°,^{419, 1321} 284.50°,⁵⁷⁴ 283.8°;¹⁰⁸ critical pressure 47.1;¹³²¹ heat of combination at constant pressure 772.17 cal.;¹²⁸⁷ heat of vaporization 8000 cal.;¹²⁸⁶ surface tension 25.78 dynes/sq. cm. at 15°,¹²⁹² 22.19,⁷⁸ 25.2 at 20°, 24.5 at 25°, 23.9 at 30°;^{529.5} parachor 241.6,⁹¹⁹ 239.1 at 20°,⁷⁸ 239.8 at 20.5°;^{903a} molar volume at 0° 107.6, at b.p. 121.5;⁶⁰⁹ molar refractivity 130.08;¹³²⁶ association factor 1.034 at 16–31°, 0.824, at 79–84°,²⁶⁴ at b.p. 1.01;^{1336c} viscosity 467 × 10⁵ at 15°,¹²⁹² 0.446,⁹¹⁹ 0.440 at 20°,^{529.5} 0.422,⁹¹⁹ 0.417 at 25°, 0.396 at 30°;^{529.5} magnetic susceptibility 707 × 10⁻⁷;^{974a} specific cohesion 6.65;^{1142, 1336c} heat of formation 33.7 cal.¹⁰⁷ See Table 1.3.
- Propyl, Pr₂S, m. -101.9°,^{1291a} -102.6°;⁸¹⁹ b. 142°,^{353, 1105, 1176, 1367} 142.8°,^{608, 819, 1319} 140.8°,³⁵⁹ 142.1–2.6°,^{46a} 141–2°,^{1376a} 141.5°,^{1124, 1318} 140.1–0.4°,^{623b} 140–3°,^{161b} 139–41°,⁹³³ b₇₄₅ 140°;¹²³⁴ d 0/4 0.8525,^{46a, 46b} d₁₇ 0.814,^{245b, 1367} d 20/4 0.8444,^{623b} 0.8377,⁸¹⁹ 0.8442,⁹³³ 0.8386,¹²³⁴ 0.8358,^{46a, 46b} 0.8302,¹⁰⁹ d 25/4 0.8332,⁸¹⁹ 0.8319,^{46a, 46b} d 61.9/4 0.8013;¹³²⁶ n 20/D 1.4787,⁸¹⁹ 1.4496,^{623b} 1.4493,¹³¹⁹ 1.4480,^{933, 1124, 1318} 1.4481,^{46a, 46b} 1.44904,¹⁰⁹ n 25/D 1.4461,⁸¹⁹ 1.4456;^{46a, 46b} magnetic susceptibility 956 × 10⁻⁷.^{974a} See Table 1.3.
- i*-Propyl, *i*-Pr₂S, m. -78.08°;⁸¹⁹ b. 120.7°,^{87, 1367} 119.8°,⁸¹⁹ 119.2–9.4°,^{46a} 118°,^{412c} 120°;^{818a, 1393} d 0/4 0.8306,^{46a, 46b} d 20/4 0.8146,⁸¹⁹ 0.8136, d 25/4 0.8092,^{46a, 46b} 0.8104,⁸¹⁹ d 41.4/4 0.7960;¹³²⁶ n 20/D 1.4388,⁸¹⁹ 1.4381, n 25/D 1.4354,^{46a, 46b} 1.4362.⁸¹⁹ See Table 1.3.
- Butyl, Bu₂S, m. -79.7°;^{1192, 1291b} b. 182°,^{353, 506, 818a, 1176, 1367} 188.1°,⁹¹⁹ 183–5°,^{976a} 186.9–7.5°,^{46a} 184–5°,^{412c} 176–85°,¹¹³⁸ 188.1–8.8°,^{623b} b₁ 20°, b_{2.8} 40°, b_{7.4} 60°, b₁₂ 70°, b_{19.7} 80°;⁸³ d 0/4 0.852,¹³⁶⁷ 0.849,^{1137a} 0.8535,^{46a, 46b} d 16/4 0.8384,¹¹³⁸ d 20/4

- 0.8386,^{46a, 46b} 0.8334,¹⁰⁹ 0.8450,^{623b} 0.8388,⁹¹⁹ d 25/4 0.8348,^{46a, 46b, 919} d 61.2/4 0.8040;¹³²⁶ n 20/D 1.4525,⁹¹⁹ 1.4529,^{46a, 46b} 1.45405,¹⁰⁹ 1.4551,^{623b} 1.4530,^{1318, 1319} n 25/D 1.4504;^{46a, 46b} surface tension 27.35 at 26.8°; parachor 398.6; viscosity 1.072, 0.995.⁹¹⁹ See Table 1.3.
- i*-Butyl, *i*-Bu₂S, m. -105.5°;¹²⁹³ b. 171°,¹³⁶⁷ 170.0°,⁹¹⁹ 168.7-9.3°,^{46a} 172°,^{818a} 170.5°,⁸⁷ 172-3°;⁵⁰⁶ d 0/4 0.8427,^{46a, 46b} d 10/4 0.8360,^{87, 1367} d 20/4 0.8285,⁹¹⁹ 0.8262, d 25/4 0.8232,^{46a, 46b} 0.8244,⁹¹⁹ d 41.2/4 0.8106;¹³²⁶ n 20/D 1.4471,⁹¹⁹ 1.4463, n 25/D 1.4439;^{46a, 46b} surface tension 25.1 at 24.6°; parachor 395.0; viscosity 0.944, 0.880.⁹¹⁹ See Table 1.3.
- s*-Butyl, (CH₃CH₂CHMe)₂S, b. 165°;^{1091, 1367} d₂₃ 0.8317.¹⁰⁹¹ See Table 1.3.
- t*-Butyl, (Me₃C)₂S, b. 148-9°,^{412a} 150°,^{1205, 1216a} b₇₁ 72°,^{660, 1095} b₈₇ 83°,¹²⁰⁵ b₁₆ 51°;⁹³⁸ n 20/D 1.4505,^{1216a} n 18/D 1.4511.⁹³⁸
- Amyl, Am₂S, m. -51.33°;¹¹⁹² b. 227.4-7.8°,^{46a} b₁₅ 108-9°;⁸⁴¹ d 0/4 0.8532, d 20/4 0.8390, d 25/4 0.8350; n 20/D 1.4556, n 25/D 1.4532.^{46a, 46b} See Table 1.3.
- i*-Amyl, (Me₂CHCH₂CH₂)₂S, b. 216°,^{64, 818a, 1367} 215.3°,⁹¹⁹ 214°,^{87, 419} 209-11°,⁴²⁷ 214.6-5.1°,^{46a} 210°,^{570b} b₄ 65-7°,⁶⁸⁹ b_{1.2} 30°, b_{3.2} 60°, b₇ 80°;⁸³ d 0/4 0.8476, d 20/4 0.8323,^{46a, 46b} 0.843,¹³⁶⁷ 0.84314,⁹³⁵ 0.8398,⁶⁸⁹ 0.8341, d 25/4 0.8308,⁹¹⁹ 0.8284,^{46a, 46b} d 60/4 0.8031;¹³²⁶ n 20/D 1.4560,⁶⁸⁹ 1.4531,⁹¹⁹ 1.44966,⁹³⁵ 1.4520, n 25/D 1.4499;^{46a, 46b} critical temperature 391.25°; heat of formation 67.5.¹⁰⁷ See Table 1.3.
- act*-Amyl, (MeCH₂CHMeCH₂)₂S, b₁₃ 95-8°; d 0/4 0.853, d 20/4 0.8360; [α] 20/D 24.52°.^{206b}
- Hexyl, (C₆H₁₃)₂S, b. 230°,⁹⁸⁴ b_{0.3} 73-4°;⁶⁸⁵ d 25/4 0.8376; n 20/D 1.4586.¹³²⁶ See Table 1.3.
- Heptyl, (C₇H₁₅)₂S, b. 298°,^{1376b} b₅ 140°.¹³⁹³ See Table 1.3.
- 4-Heptyl, (Pr₂CH)₂S, b₅ 114°.¹³⁹³
- Octyl, (C₈H₁₇)₂S, b. 310°,⁹⁰² b₁₀ 180°,⁴¹⁵ b₇ 172°,⁶⁸⁵ b₂ 150-3°;² d 17/4 0.8409,⁹⁰² d 25/4 0.8412, d 60/4 0.8164.¹³²⁶ See Table 1.3.
- i*-Octyl, (Me₂CHCH₂CH₂CH₂CH₂CH₂)₂S, b_{1.5} 125.5-7°,¹⁰⁹ b₁₄ 166.8°;^{285a} d 20/4 0.8332; n 20/D 1.46100.¹⁰⁹
- s*-Octyl, (C₆H₁₃CHMe)₂S, b. 281-3°, b₂₀ 175°,⁶⁶⁰ b₂ 135°; d 25/4 0.8382.⁶⁸¹
- Nonyl, (C₉H₁₉)₂S, b₅ 185-6°; d 20/4 0.8455; n 20/D 1.4640.¹²⁹⁷
- Decyl, (C₁₀H₂₁)₂S, m. 27°,⁶⁸⁵ 26°; b₈ 217-8°.¹²⁹⁷
- Undecyl, (C₁₁H₂₃)₂S, m. 34.8°.¹⁰⁸⁰

- Dodecyl, $(C_{12}H_{25})_2S$, m. 40.5° ,⁶²² 42.2° ,¹⁰⁸⁰ 41° ,³⁷⁷ 40° .¹¹⁰
 Tridecyl, $(C_{13}H_{27})_2S$, m. 48.2° .¹⁰⁸⁰
 Tetradecyl, $(C_{14}H_{29})_2S$, m. 50° ,⁶²² 53.8° .¹⁰⁸⁰
 Pentadecyl, $(C_{15}H_{31})_2S$, m. 59.0° .¹⁰⁸⁰
 Cetyl, $(C_{16}H_{33})_2S$, m. 57° ,¹¹⁰ 57.5° ,⁴⁴⁶ 58° ,⁶²² 58.5° ,⁵⁸ 1384 62° ,⁵⁹⁴ 63° ; ¹⁰⁸⁰ dipole moment 1.47; ¹³⁸⁴ magnetic susceptibility -401.7 .³³⁵
 Heptadecyl, $(C_{17}H_{35})_2S$, m. 66.2° .¹⁰⁸⁰
 Octadecyl, $(C_{18}H_{37})_2S$, m. 64.5° ,¹¹⁸⁰ 69° ,^{622,1080} 71° .^{1180.1}
 Cyclopentyl, $(C_5H_9)_2S$, b_{24} $129.5-30.5^\circ$,⁷⁹⁵ b_{26} 134° ; d 20/4 0.9726; n 20/D 1.5102.¹²⁹⁵
 Cyclohexyl, $(C_6H_{11})_2S$, m. -8° ; ⁸⁹¹ $b_{0.2}$ 74° ,⁹³⁸ b_8 $136-8^\circ$,¹²⁹⁵ b_{50} 180° ; ⁶⁶⁰ d 20/4 0.9687; n 18.5/D 1.5162,⁹³⁸ n 20/D 1.5146,¹²⁹⁵ n 25/D 1.50473.⁶⁶⁰
 Cycloheptyl, $(C_7H_{13})_2S$, b_{11} 174° .⁷⁹⁵
 3-Methylcycloheptyl, $(MeC_7H_{12})_2S$, b_{11} $165-8^\circ$.⁷⁹⁵

Symmetrical Unsaturated Sulfides

- Vinyl, $(CH_2:CH)_2S$, b. 83° ,^{771b} 85° ,³⁴⁶ $83.5-4^\circ$,¹¹²³ $85-6^\circ$,^{66a} $84.5-5.5^\circ$; ^{903b} d 15/4 0.9174,^{66a} 0.9125.¹¹⁷¹
 Allyl, $(CH_2:CHCH_2)_2S$, m. -83° ; ^{1291b} b. 138.6° ,²¹ $1074a$ 138.7° ,^{937a} 1105 $138.5-9.5^\circ$,⁵⁶ $138-40^\circ$,¹⁰⁴² 138° ,¹¹⁷⁶ 140° ,²⁴⁷ 312 ,⁵⁹⁶ $139-41^\circ$,⁸⁰⁴ $138-9^\circ$,¹⁹⁶ 419 137° ,^{412c} ^{771b} b_{15} $35.6-6^\circ$,²¹ b_{16} 36° ,^{771b} $b_{20.5}$ 40° ,⁸³ b_{20} 41.5° , b_{80} 71.7° , b_{200} 94.9° , b_{700} 135.5° ; ^{1074a} d_{11} 0.8544,⁴⁹⁹ d 26.8/4 0.88765; ³⁹⁰, ^{937a} n 11/D 1.4598,⁴⁹⁹ n 20/D 1.4895,¹⁰⁴² n 26.8/D 1.48770,³⁹⁰, ^{937a} critical temperature 380.38° ; ⁴¹⁹ heat of formation -16.1 cal.¹⁰⁷
 Crotyl, $(MeCH:CHCH_2)_2S$, b. $186-7^\circ$,²⁸² 186.5° ,^{1074a} b_{15} 78° ,¹¹⁷⁶ b_{20} 88.9° , b_{30} 96.7° , b_{50} 108.2° ; ^{1074a} d_0 0.9032; ²⁸² n 25/D 1.495.⁵³⁰
 2-Methylallyl, $(CH_2:CMeCH_2)_2S$, b. $172.8-3.0^\circ$; d 20/4 0.8836; n 20/D 1.4862.¹²⁶⁴
 2-Methyl-2-butenyl, $(MeCH:CMeCH_2)_2S$, b_5 $60-8^\circ$; d 26/4 0.975; n 20/D 1.530.²⁶
 Hexenyl, $(C_6H_{11})_2S$, b. $168-70^\circ$.³⁶⁰
 6,6'-Thio-bis(5-methyl-4-nonene), $(PrCH:CMeCPr)_2S$, n 20/D 1.482.²⁶
 Oleyl, $(C_{18}H_{35})_2S$, m. 45° ; $b_{0.5}$ $260-80^\circ$.^{760a}
 Cyclopentenyl, $(C_5H_7)_2S$, b_{16} 112° .¹⁹⁷
 2-Cyclohexenyl, $(C_6H_9)_2S$, b_{15} 150° ; d_{25} 1.063; n 25/D 1.5500.⁸¹⁶
 3-Cyclohexenyl, $b_{0.1}$ $81-2^\circ$; n 25/D 1.5506.⁴⁰⁹, ¹¹⁷⁶

Unsymmetrical Alkyl Sulfides

- Methyl ethyl, MeSEt m. -105.91° ,^{529.5, 819} -104.8° ;²⁶³ b. 66.7° ,^{529.5} 66.6° ,⁸¹⁹ 66° ,^{263, 419, 740, 1321, 1367, 1393} 66.9° ,^{700d} $67-8^{\circ}$,¹⁹³ 68° ,⁸⁷ 65° ,¹²⁸⁶ 64° ,^{700a} $66.4-7.4^{\circ}$,⁴¹⁹ $68-70^{\circ}$; ¹⁴⁰ d 20/4 0.8422,^{529, 819} 0.836,^{700a} d_{20} 0.837,^{700a, 1367} d 21/4 0.8369,^{263, 1321} d 25/4 0.83679,^{529.5} 0.8369,⁸¹⁹ 0.8393,¹³²⁶ d 30/4 0.83145,^{529.5} d 40.7/4 0.8203;¹³²⁶ n 20/D 1.44035,^{529.5} 1.4403, n 25/D 1.4372,⁸¹⁹ 1.43737;^{529.5} critical temperature 259.66° ; ^{419, 1321} critical pressure 41.9;¹³²¹ heat of vaporization 7250 cal.;¹²⁸⁶ surface tension 24.9 at 20° , 24.2 at 25° , 23.4 at 30° ; viscosity 0.373 at 20° , 0.354 at 25° , 0.337 at 30° .^{529.5} See Table 1.3.
- propyl, MeSPr m. -112.98° ; b. 95.6° ,⁸¹⁹ $94.5-5.6^{\circ}$; d 20/4 0.8438,¹⁰³¹ 0.8424, d 25/4 0.8345; n 20/D 1.4442,⁸¹⁹ 1.4436,¹⁰³¹ n 25/D 1.4415.⁸¹⁹
- *i*-propyl, MeSCHMe₂, m. -101.48° ; b. 84.8° ,⁸¹⁹ 94° ,¹³⁶⁷ $81-2^{\circ}$,^{57.5} $90-3^{\circ}$,¹³⁹ $93-5^{\circ}$; ⁹⁵⁰ d 20/4 0.8291, d 25/4 0.8251; n 20/D 1.4390,^{57.5} 1.4392, n 25/D 1.4362.⁸¹⁹
- butyl, b. 122.5° ; d 20/4 0.8427; n 20/D 1.4477.¹³²⁶ See Table 1.3.
- *i*-butyl, MeSCH₂CHMe₂, b. 112.5° ; d 20/4 0.8335; n 20/D 1.4433.¹³²⁶ See Table 1.3.
- *t*-butyl, MeSCMe₃, b. 99° ,¹³²⁶ $101-2^{\circ}$; ¹⁰⁹⁵ d 20/4 0.8257; n 20/D 1.4402.¹³²⁶ See Table 1.3.
- amyl, MeSA_m, b. $144.5-5.5^{\circ}$; ¹²² d. 20/4 0.843; n 20/D 1.448.⁵³⁰
- *i*-amyl, MeSCH₂CH₂CHMe₂, b. $136-8^{\circ}$.⁹⁵⁰
- *act*-amyl, MeSCH₂CHMeEt, b. $139-40^{\circ}$, b_{751} $138-9^{\circ}$; d 0/4 0.8583,^{206b} d 19/4 0.8410;^{206b, 1399} $[\alpha]$ 19/D 12.30° ,^{206b} $[\alpha]$ 20/D 13.24 .^{206a, 1399}
- hexyl, MeSC₆H₁₃, b. $170-2^{\circ}$; b_{13} $61-2^{\circ}$.¹⁹⁸
- octyl, MeSC₈H₁₇, b_{10} $87-8^{\circ}$,^{57.5} b_{17-8} $100.5-2.5^{\circ}$; n 20/D 1.4580,^{57.5} n 21/D 1.457.⁷⁴²
- *s*-octyl, MeSCHMeC₆H₁₃, b_{18} $90-1^{\circ}$,⁶⁸¹ b_{10} $80-2^{\circ}$; n 20/D 1.4561.^{57.5}
- decyl, MeSC₁₀H₂₁, b_{13} 125° .¹⁹⁸
- dodecyl, MeSC₁₂H₂₅, b_{19} $163-5^{\circ}$.⁷⁴²
- cetyl, MeSC₁₆H₃₃, m. 20.5° .⁷⁴²
- cyclopentyl, MeSC₅H₉, b. $158-9^{\circ}$.⁷⁹⁵
- cyclohexyl, MeSC₆H₁₁, b. $179-80^{\circ}$,^{158b} b_{18} $68.0-8.5^{\circ}$, b_{19} $69-72^{\circ}$; ^{57.5} n 20/D 1.4945,⁷⁷ 1.4942.^{57.5}

- 3-methylcyclohexyl, $\text{MeSC}_6\text{H}_{10}\text{Me}$, *cis* b. 184° ,^{915a} 185° ; d_{25} 0.9227,⁹¹⁶ 0.923; ^{915a} n 25/D 1.4825; ^{915a}, ⁹¹⁶ *trans* b. 186° ; d_{25} 0.9220; n 25/D 1.4845.^{915a}, ⁹¹⁶
- 2-decahydronaphthalene, $\text{MeSC}_{10}\text{H}_{17}$, b. 240° ; d_{25} 0.964; n 25/D 1.4988.^{915a}, ⁹¹⁶
- Ethyl propyl, EtSPr , m. -117.04° ; b. 118.5° ,⁸¹⁹ $115-7^\circ$,¹⁹³, ³⁰⁶, ⁶⁴⁷, ⁶⁶⁰, ^{1236b} 116° ,¹³⁶⁷ $110-2^\circ$,^{818a} 118° ; ⁵⁹⁴ d 15/4 0.84901,³⁰⁶ d 20/4 0.8370,⁸¹⁹ 0.84448,³⁰⁶ 0.8391,⁶⁴⁷ d 25/4 0.8324; ⁸¹⁹ n 20/D 1.4471,⁶⁴⁷ 1.4461, n 25/D 1.4434.⁸¹⁹
- *i*-propyl, m. -122.19° ; b. 107.3° ,⁸¹⁹ 103.4 ,⁶⁶⁰, ^{1236b} 103° ; ¹³⁶⁷ d 20/4 0.8246, d 25/4 0.8199; n 20/D 1.4407, n 25/D 1.4382.⁸¹⁹
- butyl, EtSBu , m. -95.13° ; b. 144.2° ,⁸¹⁹ $144-5^\circ$,^{1366b} $143-5^\circ$,⁴⁸⁴ b_{30} $50-2^\circ$; ⁵⁴⁶ d 0/4 0.8760,^{1366b} d 20/4 0.8376, d 25/4 0.8332,⁸¹⁹ 0.8549,^{1366b} 0.8328, d 41.4/4 0.8200; ¹³²⁶ n 20/D 1.4491, n 25/D 1.4463.⁸¹⁹ See Table 1.3.
- *i*-Butyl, $\text{EtSCH}_2\text{CHMe}_2$, b. 134.2° ,⁸¹⁹ $132-4^\circ$,¹³⁸⁶ $129-32^\circ$,^{1326b} $133.5-4.7^\circ$; ⁶⁴⁷ d 15/4 0.8337,¹³⁸⁶ d 20/4 0.8306,⁸¹⁹ 0.8321,⁶⁴⁷ d 25/4 0.8261; ⁸¹⁹ n 15/D 1.44677,¹³⁸⁶ n 20/D 1.4452,⁶⁴⁷ 1.4450, n 25/D 1.4424.⁸¹⁹
- *s*-butyl, EtSCHMeEt , b. 133.6° ,⁸¹⁹ $130-1.5^\circ$; ^{13a} d 20/4 0.8353, d 25/4 0.8307; n 20/D 1.4477, n 25/D 1.4451.⁸¹⁹
- *t*-butyl, EtSCMe_3 , m. -88.95° ; b. 120.4° ,⁸¹⁹ $116.5-8.5^\circ$,^{13a} 119.5° ,¹²⁰⁵, ¹³²⁶ b_{109} $56-7^\circ$; ¹⁰⁹⁵ d 20/4 0.8206, d 25/4 0.8161,⁸¹⁹ 0.8158, d 41.3/4 0.8020; ¹³²⁶ n 20/D 1.4417, n 25/D 1.4390.⁸¹⁹ See Table 1.3.
- *i*-amyl, $\text{EtSCH}_2\text{CH}_2\text{CHMe}_2$, b. $160-0.4^\circ$,⁸⁷ $158-61^\circ$,³ $158-9^\circ$,⁶⁴⁷, ^{818a}, ^{1137a} $157-9^\circ$; ^{1137d}, ¹¹⁴⁰ d 0/4 0.852,^{1137a} d 20/4 0.8349; n 20/D 1.4495.⁶⁴⁷
- *act*-amyl, $\text{EtSCH}_2\text{CHMeEt}$, b_{738} 159° ; d 19/4 0.836,^{206b} 0.8381; ¹³⁹⁹ $[\alpha]$ 19/D 14.71° ,^{206a}, ¹³⁹⁹ 13.75° .^{206b}
- *s-i*-amyl, EtSCHMeCHMe_2 , b_{751} $150-2.5^\circ$; n 20/D 1.4527.⁶⁴⁷
- hexyl, $\text{EtSC}_6\text{H}_{13}$, b. $170-6^\circ$.^{1236b}
- heptyl, $\text{EtSC}_7\text{H}_{15}$, b. $189-93^\circ$, d_{20} 0.871, n 20/D 1.4518.³
- octyl, $\text{EtSC}_8\text{H}_{17}$, b_{100} $154-6^\circ$,⁶⁶⁰ b_{11} $102-3^\circ$,⁴¹⁵, ⁶⁶⁰ 103° ; n 20/D 1.4570.^{57.5}
- *s*-octyl, $\text{EtSCHMeC}_6\text{H}_{13}$, b_{100} $145-7^\circ$,⁶⁶⁰ b_{17} $99-100^\circ$,⁶⁸¹ $b_{2.5}$ $66-9^\circ$, b_8 $87-9^\circ$, b_9 $85-90^\circ$; n 20/D 1.4563, 1.4560.^{57.5}
- dodecyl, $\text{EtSC}_{12}\text{H}_{25}$, m. -5° ; b_{18} $167-71^\circ$.¹⁶²
- cetyl, $\text{EtSC}_{16}\text{H}_{33}$, m. 32.1° ,¹⁰⁸⁰ 19° ,¹⁶² 18° ; ⁵⁹⁴ b_{12} $201-5^\circ$.¹⁶²

- cyclopentyl, EtSC_5H_9 , b. $176-7^\circ$.⁷⁹⁵
- cyclohexyl, $\text{EtSC}_6\text{H}_{11}$, b_{10} $68-70^\circ$, $70-4^\circ$; n 20/D 1.4908, 1.4904.^{57.5}
- α -furfuryl, b. $90.5-1^\circ$; d 20/4 1.04958; n 20/D 1.5140.⁶⁹⁶
- Propyl *i*-propyl, PrSCHMe_2 , b. 132.1° , ⁸¹⁹ 132° ; ¹⁹⁶ d 20/4 0.8269, d 25/4 0.8225; n 20/D 1.4440, n 25/D 1.4414.⁸¹⁹
- butyl, PrSBu , b. $153-5^\circ$,¹⁹³ $157-8$.^{152.5}
- *i*-amyl, $\text{PrSCH}_2\text{CH}_2\text{CHMe}_2$, b. $180-2^\circ$; d_{20} 0.851; n 20/D 1.4495.³
- octyl, $\text{PrSC}_8\text{H}_{17}$, $b_{0.03}$ $45-7^\circ$.⁶⁸³
- *s*-octyl, $\text{PrSCHMeC}_6\text{H}_{13}$, b_{15} 111° ; n 20/D 1.4573.^{57.5}
- i*-Propyl butyl, $\text{Me}_2\text{CHSC}_4\text{H}_9$, b_{61} $78.5-9^\circ$; n 20/D 1.4479.³⁷⁷
- *t*-butyl, $\text{Me}_2\text{CHSCMe}_3$, b. $129-31^\circ$, b_{31-2} $40-2^\circ$.¹²⁰⁵
- amyl, Me_2CHSAm , b. 177° ; d 20/4 0.834.⁵³⁰
- *t*-amyl, $\text{Me}_2\text{CHSCMe}_2\text{Et}$, b. $170-5^\circ$.^{13a}
- dodecyl, $\text{Me}_2\text{CHSC}_{12}\text{H}_{25}$, $b_{0.5}$ 109° .^{1190d}
- Butyl *i*-butyl, $\text{BuSCH}_2\text{CHMe}_2$, b. $117-8^\circ$,¹⁹⁶ $178-9^\circ$; d 20/4 0.8341; n 20/D 1.4999.⁶⁴⁷
- heptyl, $\text{BuSC}_7\text{H}_{15}$, b_{27} 135° .^{1190d}
- *s*-octyl, $\text{BuSCHMeC}_6\text{H}_{13}$, b_8 113° , $114-6^\circ$; n 20/D 1.4590.^{57.5}
- dodecyl, $\text{BuSC}_{12}\text{H}_{25}$, b_4 $108-15^\circ$,¹⁰⁷⁵ b_9 $168-71^\circ$; n 20/D 1.4648.³⁷⁷
- octadecyl, $\text{BuSC}_{18}\text{H}_{37}$, m. 32.0° ,¹⁰⁸⁰ 30° .¹¹⁸⁰
- cyclohexyl, $\text{BuSC}_6\text{H}_{11}$, b_{10} $101-3^\circ$; n 20/D 1.4864.^{57.5}
- i*-Butyl hexyl, $\text{Me}_2\text{CHCH}_2\text{SC}_6\text{H}_{13}$, n 20/D 1.4569.⁵⁴⁴
- i*-Amyl cyclohexyl, $\text{Me}_2\text{CHCH}_2\text{CH}_2\text{SC}_6\text{H}_{11}$, b_{12} 120° ,^{336a} $b_{0.1}$ $58-60^\circ$; n 20/D 1.4860.^{130b}
- 4-methylcyclohexyl, $\text{Me}_2\text{CHCH}_2\text{CH}_2\text{SC}_6\text{H}_{10}\text{Me}$, b_{10} 124° .^{336a}
- 2-Octyl octadecyl, $\text{C}_8\text{H}_{17}\text{SC}_{18}\text{H}_{37}$, m. 24° .⁶⁸⁰
- Decyl octadecyl, $\text{C}_{10}\text{H}_{21}\text{SC}_{18}\text{H}_{37}$, m. 48.0° ,¹⁰⁸⁰ 45° .¹¹⁸⁰
- cyclopentyl, $\text{C}_{10}\text{H}_{21}\text{SC}_5\text{H}_9$, b_2 158° ; d 20/4 0.8833; n 20/D 1.4786.¹²⁹⁶
- cyclohexyl, $\text{C}_{10}\text{H}_{21}\text{SC}_6\text{H}_{11}$, b_2 $164-5^\circ$; d 20/4 0.8846; n 20/D 1.4820.¹²⁹⁶
- Dodecyl octadecyl, $\text{C}_{12}\text{H}_{25}\text{SC}_{18}\text{H}_{37}$, m. 53.8° ,¹⁰⁸⁰ 31.5° .¹¹⁸⁰
- Cetyl cyclohexyl, $\text{C}_{16}\text{H}_{33}\text{SC}_6\text{H}_{11}$, $b_{1.5}$ $192-8^\circ$; n 20/D 1.4793.^{356a}
- Cyclopentyl cyclohexyl, $\text{C}_5\text{H}_9\text{SC}_6\text{H}_{11}$, b_3 $119-20^\circ$; d 20/4 0.9692; n 20/D 1.5118.¹²⁹⁶

Unsaturated Unsymmetrical Sulfides

- Methyl vinyl, MeSCH:CH_2 , b. 67.3° ,¹⁰³⁰ $69-70^\circ$,²⁸¹ $66.4-6.8^\circ$,¹⁰³³
 $b_{745} 67.0^\circ$; d 20/4 0.9026; n 20/D 1.4845,¹⁰³¹ 1.4835,¹⁰³³ n
 25/D 1.4802.¹⁰³⁰
- allyl, $\text{MeSCH}_2\text{CH:CH}_2$, b. $92-2.5^\circ$,⁷³⁸ $92.2-2.4^\circ$,¹⁰³¹ $91-3^\circ$; ⁹⁵⁰
 d 20/4 0.8767; n 20/D 1.4712.¹⁰³¹
- 2-methylallyl, $\text{MeSCH}_2\text{CMe:CH}_2$, b. $113.0-3.2^\circ$; n 20/D
 1.4712.⁷⁷
- 3-isoprenyl, $\text{MeSC(CMe:CH}_2\text{):CH}_2$, $b_{19} 46-8^\circ$.⁴⁹
- 6-hexenyl-2, $\text{MeSCH}_2\text{CH}_2\text{CH}_2\text{CH:CHMe}$, $b_{748} 161-3^\circ$, b_{15}
 $51.5-2^\circ$; d 18/4 0.8985; n 18/D 1.4774.⁵¹⁷
- 2-cyclohexenyl, MeSC_6H_9 , b. 155° ; $d_{25} 0.960$; n 25/D
 1.4694.⁹¹⁸
- Ethyl vinyl, EtSCH:CH_2 , b. $91.5-2^\circ$,⁷⁹³ 91° ,^{633a}, ^{687a} $91.9-$
 2.2° ; ^{1035.5} $d_{14} 1.887$,^{1236b} $d_{20} 0.8756$,^{1035.5} d 20/4 0.873,⁵³⁰ d_{25}
 0.8691 ; ⁷⁹³ n 20/D 1.4756,^{1035.5} n 25/D 1.4631.⁷⁹³
- allyl, $\text{EtSCH}_2\text{CH:CH}_2$, b. $115-6^\circ$; d 20/4 0.8676.⁵³⁰
- propenyl, EtSCH:CHMe , b. $125-30^\circ$.⁷⁹³
- methylvinyl, EtSCMe:CH_2 , b. $109-10^\circ$.^{41a}
- heptenyl, $\text{EtSCH:CH(CH}_2\text{)}_4\text{Me}$, b. $196-202^\circ$.⁷⁹³
- oleyl, $\text{EtSC}_{18}\text{H}_{35}$, $b_{0.3} 191-5^\circ$.^{760a}
- 3-isoprenyl, $\text{EtSC(CMe:CH}_2\text{):CH}_2$, dimer $b_{4-5} 115-25^\circ$.⁴⁹
- Propyl allyl, $\text{PrSCH}_2\text{CH:CH}_2$, b. $138-40^\circ$.⁴⁰⁹
- 3-isoprenyl, $\text{PrSC(CMe:CH}_2\text{):CH}_2$, dimer $b_{4-5} 120-30^\circ$.⁴⁹
- i*-Propyl 3-isoprenyl, $i\text{-PrSC(CMe:CH}_2\text{):CH}_2$, $b_{20} 65-8^\circ$; dimer
 $b_{5-6} 130-40^\circ$.⁴⁹
- Butyl crotyl, $\text{BuSCH}_2\text{CH:CHMe}$, $b_{738} 186^\circ$; $d_{25} 0.8551$, n 25/D
 1.4703.⁷⁹⁹
- 1-methallyl, BuSCHMeCH:CH_2 , $b_{740} 170.5-1.5^\circ$; $d_{25} 0.8758$;
 n 25/D 1.4623.⁷⁹⁹
- methallyl, $\text{BuSCH}_2\text{CMe:CH}_2$, $b_{42} 90.4^\circ$; d 20/4 0.8592, d
 25/4 0.8546; n 20/D 1.4685, n 25/D 1.4656.¹¹⁶⁹
- BuSCH:CH:C:CH , $b_8 80-2^\circ$.^{286.5}
- t*-Butyl 3-isoprenyl, $t\text{-BuSC(:CH}_2\text{)CMe:CH}_2$, b. $172-4^\circ$.⁴⁹
- Amyl dihydronordicyclopentadienyl, $b_3 135-40^\circ$.²²⁸
- i*-Amyl vinyl, $i\text{-AmSCH:CH}_2$, $b_4 30-2^\circ$; d 20/4 0.8656; n 20/D
 1.4715.⁶⁸⁹
- t*-Amyl 3-methylcrotyl, $t\text{-AmSCH}_2\text{CH:CMe}_2$, $b_{3.5} 60-5^\circ$; d 20/4
 0.8913; n 20/D 1.48615.²¹⁴

Cyclohexyl cyclohexenyl, $C_6H_{11}SC_6H_9$, $b_{0.1}$ 69–70°; ⁴⁰⁹, ¹¹⁷⁶ n 19/D 1.5317.⁴⁰⁹

Phenyl Alkyl Sulfides, PhSR

Methyl, PhSMe, b. 188°; ³²⁶ 189–90°; ^{412c}, ^{1261a} 187–8°; ⁹⁵⁰ 187°; ³⁵³ 195°; ¹⁷⁰ 187–90°; ¹⁸³, ^{1261d} b_{757} 193.5–5.0°; ^{1031.3} b_{770} 189.8–90.2°; ⁶¹¹ b_8 58–60°; ¹²⁴⁴ $b_{16.5}$ 80.0°; ⁷⁷ b_{18} 82°; ⁶⁷ d_{11} 1.117, d_{15} 1.104; ^{1261d} d 20/4 1.053; ⁶⁷ 1.0576; ¹⁸³ d 25/4 1.0533; ¹²⁴⁴ n 20/D 1.58693; ¹⁸³ 1.5835; ⁶⁷ n 25/D 1.5832; ¹²⁴⁴ 1.5835; ^{1031.3} n 26.5/D 1.5826.^{1261d}

Ethyl, PhSEt, b. 205°; ⁸⁷ 204°; ¹²¹¹ 204.5–5.5°; ¹³⁸⁶ 200–202°; ^{556.5} 202–5°; ⁴⁸⁴ 203–6°; ^{1261d} b_{743} 204°; ⁸⁷ b_{20} 94°; ^{412c} b_{14} 86–7° 201.5 d 10/4 1.0312; ⁸⁷ d_{10} 1.031; ³⁵³ d 15/4 1.024; ¹³⁸⁶ n 15/D 1.5701, n 22.5/D 1.5662.^{1261d}

Propyl, PhSPr, b. 219–20°; ⁶⁴⁸ 215–20°; ^{152.5} 89–90°; ¹²⁶⁷ d 20/4 0.9995; n 20/D 1.5571.⁶⁴⁸

i-Propyl, PhSCHMe₂, b. 207–8°; ⁶⁴⁸ b_{16} 92–4°; ¹³⁹ b_{20} 97°; ^{412c} d 20/4 0.9855; n 20/D 1.5468.⁶⁴⁸

Butyl, PhSBu, b_4 94.5–7°; ⁶⁴⁸ 96–8°; ⁴⁹⁵ b_{12} 137–9°; ⁸⁴¹ d 20/4 0.9952; n 20/D 1.5463; ⁶⁴⁸ 1.5458.⁴⁹⁵

i-Butyl, PhSCH₂CHMe₂, b_{12} 126–7°; ⁸⁴¹ b_{13} 107–8°; d 20/4 0.9803; n 20/D 1.5430.⁶⁴⁸

s-Butyl, PhSCHMeEt, b_1 63.5°; ³²⁵ b_4 90–1°; ⁶⁴⁸ b_{25} 104–5°; ¹²⁷⁴ d 20/4 0.9789; ⁶⁴⁸ d 25/4 0.9732; ³²⁵ n 20/D 1.5425; ⁶⁴⁸ n 25/4 1.5395.³²⁵

t-Butyl, PhSCMe₃, b_{19} 100°; ^{412c} b_5 73°; d 20/4 0.9895; n 20/D 1.5335.⁶⁴⁸

Amyl, PhSAme, b_8 117–8°; $b_{4.5}$ 93–4°; d 20/4 0.9714; n 20/D 1.5396.⁶⁴⁸

i-Amyl, PhSCH₂CH₂CHMe₂, b. 240–2°; ²³⁷ b_6 100–0.5°; d 20/4 0.9681; n 20/D 1.5380.⁶⁴⁸

s-Amyl, PhSCHMePr, $b_{4.5}$ 93–4°; d 20/4 0.9642; n 20/D 1.5357.⁶⁴⁸

act-Amyl, PhSCH₂CHMeEt, $b_{4.5}$ 99–100°; d 20/4 0.9783; n 20/D 1.5408.⁶⁴⁸

Diethylmethyl, PhSCHEt₂, b_9 107–7.5°; d 20/4 0.9738; n 20/D 1.5385.⁶⁴⁸

s-i-Amyl, PhSCHMeCHMe₂, b_5 99–100°; d 20/4 0.9794; n 20/D 1.5394.⁶⁴⁸

- t*-Amyl, PhSCMe_2Et , b_6 91–1.3°; d 20/4 0.9679; n 20/D 1.5351.⁶⁴⁸
 Octyl, $\text{PhSC}_8\text{H}_{17}$, b_{18} 175°.^{233a}
s-Octyl, $\text{PhSCHMeC}_6\text{H}_{13}$, b_{20} 178°; d 17/4 0.99; n 18/D 1.5256.⁶⁸¹
 Nonyl, $\text{PhSC}_9\text{H}_{19}$, m . 21.9°; d 25/4 0.924.¹⁴⁴
 Decyl, $\text{PhSC}_{10}\text{H}_{21}$, m . 21°; b_5 170–1°; d 20/4 0.9341, n 24/D 1.5213.¹²⁹⁶
 Nonyl to eicosyl. See Table 5.3.
 Cetyl, $\text{PhSC}_{16}\text{H}_{33}$, m . 50.8°, ¹⁰⁸⁰ 49.4°; ¹⁴⁴ b_2 201–2°; ^{356a} d 50/4 0.889.¹⁴⁴
 Octadecyl, $\text{PhSC}_{18}\text{H}_{37}$, m . 56.5°, ¹¹⁸⁰ 57°.¹⁰⁸⁰
 Cyclopentyl, PhSC_5H_9 , b_{13} 139.5°; d 20/4 1.0571; n 20/D 1.5740.¹²⁹⁶
 1-Methylcyclopentyl, b_4 115°.⁸¹³
 Cyclohexyl, $\text{PhSC}_6\text{H}_{11}$, b_{11} 145°, ⁴⁸⁷ b_{13} 126°, ²²⁰ $b_{0.1}$ 108°; ^{336a} d 20/4 1.0013; n 20/D 1.5663.⁴⁸⁷
 2-Methylcyclohexyl, $\text{PhSC}_6\text{H}_{10}\text{Me}$, $b_{0.1}$ 120°, ^{336a} $b_{0.05}$ 108°.^{336b}
 Methylcyclohexyl, $\text{PhSC}_6\text{H}_{10}\text{Me}$, b_{17} 150°; n 17/D 1.5626.²³⁷
 Vinyl, PhSCH:CH_2 , b . 200–1°, ^{194a} 194–5°, ¹⁰³⁷ b_{745} 198°, ^{1031.5} b_8 55°, ²⁰⁵ b_{15} 84.5–5°, b_{25} 94–4.5°, b_{45} 102°, b_{48} 106°; d 20/4 1.0386; n 25/D 1.5878, MR_D 44.12.^{1031.5}
 α -Methylvinyl, PhSCMe:CH_2 , b . 207–8°.^{41a}
 Allyl, $\text{PhSCH}_2\text{CH:CH}_2$, b . 215–8°, b_{25} 104–6°, ⁶²⁶ $b_{1.5}$ 59–60°, ¹²⁶⁷ $b_{0.43}$ 48–9°; ³²⁵ d 20/4 1.0275, ¹²⁶⁷ d 25/4 1.0220; ³²⁵ n 20/D 1.5760, ¹²⁶⁷ 1.4772, ⁶²⁶ n 25/D 1.5732.³²⁵
 Propenyl, PhSCH:CHMe , n 20/D 1.5850.¹²⁶⁷
 1-Methylallyl, PhSCHMeCH:CH_2 , $b_{1.2}$ 56–8°; d 25/4 0.9876; n 25/D 1.5546.³²⁵
 Crotyl, $\text{PhSCH}_2\text{CH:CHMe}$, $b_{1.3}$ 69.6–70°; d 25/4 1.0045; n 25/D 1.5680.³²⁵
 3-Isoprenyl, $\text{PhSC(:CH}_2\text{)CMe:CH}_2$, b_4 82–6°.⁴⁹
 Dihydronordicyclopentadienyl, b_2 157°.²²⁸
 2-Cyclopentenyl, PhSC_5H_7 , b_{15} 190°; d_{25} 1.222.⁹¹⁶
 2-Cyclohexenyl, PhSC_6H_9 , m . 57°; b_{15} 198°.⁹¹⁶

Benzyl Alkyl Sulfides, PhCH_2SR

- Methyl, PhCH_2SMe , b . 195–8°, ⁹⁵⁰ $b_{1.8}$ 52–4°, ⁷⁷³ b_{11} 87–8°, ^{802c} b_{12} 89–90°, ¹³⁹ 91–2°, ¹⁹³ 90–1°, ^{228.5} 97–8°; ¹⁴⁰ n 25/D 1.5550.⁷⁷³
 Ethyl, PhCH_2SEt , b . 220–3°, ⁴⁸⁴ 222–3°, ^{135a} 214–6°, ^{832b} 218–24°, ^{1236b} 218–20°, ³ b_{25} 116–7°, ^{412c} b_{13} 98–9°.^{228.5}
 Propyl, PhCH_2SPr , b_{14} 112°.^{228.5}

- i*-Propyl, $\text{PhCH}_2\text{SCHMe}_2$, b_{14} 99–104°.^{228.5}
 Butyl, PhCH_2SBu , b_{14} 123°.^{228.5}
i-Butyl, $\text{PhCH}_2\text{SCH}_2\text{CHMe}_2$, b. 243–4°,³ b_{13} 118°;^{228.5} d 20/4 0.968; n 20/D 1.4912.³
t-Butyl, $\text{PhCH}_2\text{SCMe}_3$, m. 49°; b_{15} 115–6°.¹⁰⁹⁵
 Amyl, PhCH_2SAm , b_{14} 146–7°.^{228.5}
i-Amyl, $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{CHMe}_2$, b_{14} 135–6°.^{228.5}
 Decyl, $\text{PhCH}_2\text{SC}_{10}\text{H}_{21}$, b_{3-4} 142–4°.¹⁰⁷⁵
 Cyclohexyl, $\text{PhCH}_2\text{SC}_6\text{H}_{11}$, $b_{0.7}$ 108–9°,^{1349a} b_3 126°,²²⁰ b_{14} 162–6°;^{228.5} d_{20} 1.0218; n 20/D 1.5564.^{1349a}
 Vinyl, $\text{PhCH}_2\text{SCH}:\text{CH}_2$, b. 216–9°.¹⁰³⁷
 α -Methylvinyl, $\text{PhCH}_2\text{SCMe}:\text{CH}_2$, b. 225°.^{41b}
 Allyl, $\text{PhCH}_2\text{SCH}_2\text{CH}:\text{CH}_2$, b_{12} 121–2°,¹⁹³ b_{13} 110–5°,⁷¹⁵ 117–8°,⁵³ b_{14} 115–6°,^{228.5} b_5 81–4°, b_2 77–9°; d 20/4 1.0074.
 Oleyl, $\text{PhCH}_2\text{SC}_{18}\text{H}_{35}$, $b_{0.2}$ 250°; n 19/D 1.5021.^{760a}
 Cholesteryl, m. 98.5°.¹³³⁴

Other Aryl Alkyl Sulfides, ArSR

- 1-Phenylethyl methyl, PhCHMeSMe , b_4 70°; n 20/D 1.5497.^{57.5}
 — ethyl, PhCHMeSEt , b_3 73–4°, $b_{3.4}$ 79–81°; n 20/D 1.5397.^{57.5}
 — butyl, PhCHMeSBu , $b_{0.25}$ 69–71°; n 20/D 1.5272.^{57.5}
 2-Phenylethyl methyl, $\text{PhCH}_2\text{CH}_2\text{SMe}$, b_4 80–3°,^{57.5} b_{12} 111°;¹⁹⁸ n 20/D 1.5513.^{57.5}
 — ethyl, $\text{PhCH}_2\text{CH}_2\text{SEt}$, b_3 92–4°,^{57.5} b_4 96°;^{412a} n 20/D 1.5420.^{57.5}
 2-(2,4,6-Trinitrophenyl)ethyl methyl, $(\text{O}_2\text{N})_3\text{C}_6\text{H}_2\text{CH}_2\text{CH}_2\text{SMe}$, m. 86°.²⁷²
o-Tolyl ethyl, $\text{MeC}_6\text{H}_4\text{SEt}$, b. 120°.¹⁰⁴⁴
 — octadecyl, $\text{MeC}_6\text{H}_4\text{SC}_{18}\text{H}_{37}$, m. 39°.¹¹⁸⁰
m-Tolyl methyl, m. 108°;⁹⁵⁰ b_{31} 110°; n 24/D 1.5736.¹²⁶⁵
 — ethyl, $\text{MeC}_6\text{H}_4\text{SEt}$, b_{735} 216–9°; d_{25} 0.9947; n 15/D 1.5610.⁶⁴¹
p-Tolyl methyl, $\text{MeC}_6\text{H}_4\text{SMe}$, b_{763} 211–2°,⁶¹¹ b_{747} 209°,^{44a, 183} b_{20} 104–5°,^{484, 1346} b_{31} 94°; d 15/4 1.0302,^{44a} d 16/4 1.0302, d 20/4 1.026;¹⁸³ n 16/D 1.57537,^{44a, 183} n 20/D 1.573.¹⁸³
 — ethyl, $\text{MeC}_6\text{H}_4\text{SEt}$, b. 220–1°,^{961b} 218–20°,^{1261a, 1261d} 219–20°,⁴⁸⁴ b_{757} 221–20°,^{44b} b_{34} 117–9°,¹³⁴⁶ b_{20} 122–5°,⁴⁸⁴ b_9 91–2°,^{377a} b_2 101–3°,⁴⁸⁴ b_{15} 105°;^{44b} $d_{17.5}$ 1.0016;^{961b} n 20/D 1.5568.^{377a}
 — propyl, $\text{MeC}_6\text{H}_4\text{SPr}$, b_{15} 120°; d 20/4 0.9755.⁴⁸⁴
 — *i*-propyl, $\text{MeC}_6\text{H}_4\text{SCHMe}_2$, b_{750} 228°, b_{14} 110°.^{44b}

- butyl, $\text{MeC}_6\text{H}_4\text{SBu}$, b. $249-50^\circ$, b_{15} $135-8^\circ$,⁴⁸⁴ b_{18} $142-5^\circ$,¹¹⁹⁴ b_9 $120-2^\circ$; ^{377a} d 20/4 0.9615; ⁴⁸⁴ n 20/D 1.5408.^{377a}
- *s*-butyl, $\text{MeC}_6\text{H}_4\text{SCHMeEt}$, b_{22} $135-8^\circ$.¹²⁷⁴
- *i*-amyl, $\text{MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{CHMe}_2$, b. 253° , b_{17} 120° ,²³⁷ b_{16} 150° .⁴⁸⁵
- dodecyl, $\text{MeC}_6\text{H}_4\text{SC}_{12}\text{H}_{25}$, m. 31.5° .³⁷⁷
- octadecyl, $\text{MeC}_6\text{H}_4\text{SC}_{18}\text{H}_{37}$, m. 53° .¹¹⁸⁰
- methylcyclohexyl, $\text{MeC}_6\text{H}_4\text{SC}_6\text{H}_{10}\text{Me}$, b_{17} $168-70^\circ$; n 17/D 1.5570.²³⁷
- allyl, $\text{MeC}_6\text{H}_4\text{SCH}_2\text{CH}:\text{CH}_2$, b_{25} $123-7^\circ$,⁶²⁶ b_2 $77-9^\circ$, b_5 $81-4^\circ$; d 20/4 1.0044; ⁴⁸⁹ n 21/D 1.5711.⁶²⁶
- for undecyl to eicosyl *see* Table 5.3.
- o*-Ethylphenyl methyl $\text{EtC}_6\text{H}_4\text{SMe}$, b_{773} $228.2-8.6^\circ$; d 20/4 1.0253; n 20/D 1.57083.⁴⁴⁵
- p*-Ethylphenyl methyl, $\text{EtC}_6\text{H}_4\text{SMe}$, b. 212° ,²³⁹ b_{15} 101° ; ^{244a} d_{17} 1.007; n 17/D 1.5263.^{244a}
- 2,4-Dimethylphenyl methyl, $\text{Me}_2\text{C}_6\text{H}_3\text{SMe}$, d 17.5/4 1.0211, d 20/4 1.019.⁷³⁷
- 3,5-Dimethylphenyl methyl, $\text{Me}_2\text{C}_6\text{H}_3\text{SMe}$, b_{24} 127° ,⁴³ b_{15} 120° .^{201.5}
- 2,4,6-Trimethylphenyl methyl, $\text{Me}_3\text{C}_6\text{H}_2\text{SMe}$, b_{14} $112-3^\circ$.^{412b}
- p*-Vinylphenyl methyl, $\text{CH}_2:\text{CHC}_6\text{H}_4\text{SMe}$, b_3 $83-5^\circ$; d 25/4 1.0384; n 20/D 1.6186.⁴⁷
- p*-(α -Methylvinyl)phenyl methyl, $\text{H}_2\text{C}:\text{CMeC}_6\text{H}_4\text{SMe}$, m. 51° ; b_{1-2} 85° .⁴⁷
- 4-Biphenyl methyl, $\text{PhC}_6\text{H}_4\text{SMe}$, m. 108° .⁹⁵⁰
- ethyl, $\text{PhC}_6\text{H}_4\text{SEt}$, m. 75° .^{1266b}
- 4-Biphenyl sulfides. *See* Table 6.3 for others.
- Diphenylmethyl methyl, Ph_2CHSMe , b_1 $108-11^\circ$.⁷⁰²
- m*-Diphenylmethylphenyl methyl, $\text{Ph}_2\text{CHC}_6\text{H}_4\text{SMe}$, m. 49.5° ; d 90/4 1.0253; n 90/D 1.61352.¹⁸¹
- o*-Methylbenzyl methyl, $\text{MeC}_6\text{H}_4\text{CH}_2\text{SMe}$, b_1 $67-8^\circ$; n 25/D 1.5599.⁵⁵⁷
- allyl, $\text{MeC}_6\text{H}_4\text{CH}_2\text{SCH}_2\text{CH}:\text{CH}_2$, $b_{0.3}$ 86° .⁵⁰
- m*-Methylbenzyl allyl, $\text{MeC}_6\text{H}_4\text{CH}_2\text{SCH}_2\text{CH}:\text{CH}_2$, $b_{0.4}$ 82° .⁵⁰
- p*-Methylbenzyl allyl, $\text{MeC}_6\text{H}_4\text{CH}_2\text{SCH}_2\text{CH}:\text{CH}_2$, $b_{0.2}$ 82° .⁵⁰
- α -Naphthyl methyl, $\text{C}_{10}\text{H}_7\text{SMe}$, b_{20} $166-8^\circ$.^{1261a}
- ethyl, $\text{C}_{10}\text{H}_7\text{SEt}$, b_{25} $175-6^\circ$,^{1261a} b_{15} $167-7.5^\circ$,⁷²⁷ b_5 $152-3^\circ$; ^{556.5} d 0/4 1.1198, d 50/4 1.0797.⁷²⁷
- decyl, $\text{C}_{10}\text{H}_7\text{SC}_{10}\text{H}_{21}$, b_{7-8} $234-5^\circ$; d 20/4 0.9893; n 20/D 1.5714.¹²⁹⁶

- β -Naphthyl methyl, $C_{10}H_7SMe$, m. 64° ,⁶⁷⁵ 60° ; b_{15} 173° .¹²¹⁹
 — ethyl, $C_{10}H_7SEt$, m. 16° ; ^{675, 727} b_{15} 170.5° .^{556.5, 675, 727}
 — hexyl, $C_{10}H_7SC_6H_{13}$, b_{20} 160° .¹⁰⁵⁶
 — heptyl, $C_{10}H_7SC_7H_{15}$, m. 34° ,¹⁰⁵⁶ 32.4° .¹⁰⁸⁰
 — octyl, $C_{10}H_7SC_8H_{17}$, m. 56° ,^{356a} 61° ; ¹⁰⁸⁰ n 50/D 1.4937.^{356a}
 — vinyl, $C_{10}H_7SCH:CH_2$, b_{2-3} $125-30^\circ$.^{637a}
 1-1,2,3,4-Tetrahydronaphthyl methyl, $C_{10}H_{11}SMe$, b_{12} $158-60^\circ$.¹⁹²
 2-1,2,3,4-Tetrahydronaphthyl methyl, $C_{10}H_{11}SMe$, b_{10} $158-9^\circ$,¹⁹²
 b_{12} 155° ; d_{20} 1.0711; n 20/D 1.5290.^{1294.5}
 — nonyl, $C_{10}H_{11}SC_9H_{19}$, b_4 $218.5-9.5^\circ$; d_{20} 0.9671; n 20/D
 1.5370.^{1294.5}
 — cyclohexyl, $C_{10}H_{11}SC_6H_{11}$, b_3 $187.5-8.5^\circ$; d_{20} 1.0543; n 20/D
 1.5800.^{1294.5}
 β -(1-Methyl)naphthyl methyl, $MeC_{10}H_8SMe$, m. 48° .^{238.5}
 β -(6-Ethyl)naphthyl methyl, $EtC_{10}H_8SMe$, m. 48° ; b_{15} 185° .^{238.5}
 α -Tetralinyl methyl, $C_{10}H_{11}SMe$, b_{12} $158-60^\circ$.¹⁹²
 β -Tetralinyl methyl, $C_{10}H_{11}SMe$, b_{10} $158-9^\circ$.¹⁹²
 3-Phenanthrenyl methyl, $C_{14}H_9SMe$, m. 100° ; b_{12} 240° .⁴²⁴
 α -9,10-Diphenylanthracenyl methyl, m. 189° .⁹⁷³
 4-Tetraphthenyl methyl, $C_{12}H_{13}SMe$, b_{10} $180-2^\circ$.^{194b}
 For more β -naphthyl alkyl sulfides see Table 6.3.

TABLE 13.3

Triphenylmethyl Alkyl Sulfides, Ph_3CSR ⁸⁸⁹

	M.p. (°C)		M.p. (°C)		M.p. (°C)
Methyl *	m. 126°	Heptyl	10°	Tridecyl	70.5°
Ethyl	129°	Octyl	63°	Myristyl	65°
Propyl	96°	Nonyl	68°	Pentadecyl	71°
		Decyl	59°	Cetyl	69°
Amyl	78°	Undecyl	68°	Heptadecyl	75°
Hexyl	59°	Dodecyl	63°	Octadecyl	73°

* m. 106° ^{943, 1323a}*Symmetrical Aryl Sulfides*

Phenyl, $PhSPh$, m. -21.5° ; ^{974b} b_{760} 296° ,^{131a, 189b} b_{765} 296° ,^{131a} b_{874}
 290° , b_{700} 291.88° ,^{1074a} $b.$ 292.5° ,^{21, 1228} 292° ,^{353, 726b, 729}
 $295-7^\circ$,¹⁸⁰ 295° ,⁸⁸⁷ 293° ,¹¹⁴⁶ 290° ,^{723a, 724} $296-7^\circ$,⁹⁸⁷ 310° ,⁶⁶¹ b_3
 126° ,^{1074a} b_7 $135-7^\circ$,^{555b} b_8 156° ,^{131a} b_9 141° ,^{412c} b_{10} 148.4° ,^{1074a}

- b_{11} 151.5°, ^{169b} 151°, ^{556.5} b_{12} 148.5–9.5°, ⁷⁵⁴ b_{15} 191–2°, ⁶⁶¹ 156.6°, ^{1074a} 165°, ^{555b} b_{16} 157–8°, ³⁶⁶ $b_{16.5}$ 150°, ^{726b} 157–8°, ^{21, 729} b_{17} 159°, ^{556.5} b_{18} 159°, ^{131b, 134} 162.5°, ^{1295.5} b_{20} 162°, ⁵⁷⁹ 157–9°, ^{555a} 164.1°, b_{30} 174.7°, ^{1074a} b_{31} 171°, ^{131a} b_{50} 189.5°; d 0/4 1.1290, ^{169b} d_0 1.1300, ^{726b, 729} d 4/4 1.1266, d_{15} 1.1185, ⁹⁸⁷ d_{20} 1.1100, ^{1295.5} 1.1160, ^{131a} d 1.119, ¹²²⁸ d 15/4 1.1166, ^{169b} $d_{15.2}$ 1.1175, ^{726b, 729} d 16/4 1.118, ⁸²⁰ d 25/4 1.1097, ¹³²⁶ 1.1093, d 30/4 1.0970, ⁹⁸⁷ 1.1040, ^{169b} d 60.1/4 1.0823, ¹³²⁶ d 100/4 1.0472; ⁹⁸⁷ n 18.5/D 1.65, ⁵⁸² n 20/D 1.6312; ^{1295.5} valency angle 113 ± 3°; ¹²⁴⁶ specific magnetic rotation 3.211. ⁹⁸⁷
- Benzyl, (PhCH₂)₂S, m . 50°, ⁴³⁶ 49°, ^{412c, 832a, 976a} 45.5°, ^{1094b} 42°; ⁹⁶³ $b_{0.3}$ 111–5°; ⁷⁷³ d_{50} 1.0712, d_{75} 1.0653, d_{100} 1.0634. ⁹⁸⁷
- Benzhydryl, (Ph₂CH)₂S, m . 66.5°. ¹¹⁵⁴
- 2-Phenylethyl, (PhCH₂CH₂)₂S, m . 90°; ^{105c} b_8 190–2°, ^{412a} b_3 143°, ^{633b} b_1 137°. ⁶⁸²
- 2-Phenyl-1-methylethyl, (PhCH₂CHMe)₂S, b_{16} 209°; d 20/4 1.072; n 24.5/D 1.5794. ⁶⁸¹
- 3-Phenylpropyl, (PhCH₂CH₂CH₂)₂S, m . 73°. ^{105c}
- 1-Phenylbutyl, (PrCHPh)₂S, m . 96°; ⁴⁰⁰ d_{20} 1.026. ⁴¹⁶
- Triphenylmethyl, (Ph₃C)₂S, m . 182°. ^{1328b}
- o*-Tolyl, (MeC₆H₄)₂S, m . 64°; ^{867, 1398} b . 285°, ¹⁰⁴⁴ b_{15} 174°, ⁸⁶⁷ b_{16} 175°. ¹³⁹⁸
- m*-Tolyl, (MeC₆H₄)₂S, b . 290°, ¹⁴⁹ b_{12} 174°, ⁸⁶⁷ b_{15} 175°, ¹¹⁸⁵ b_{23} 197°, b_{25} 200°, b_{28} 202°. ¹⁴⁹
- p*-Tolyl, m . 57.3°, ^{169b} 57°, ^{555a, 556.5, 961a, 1115, 1301b, 1398} 57.2°; ^{1113.5} b . 300°, ^{961a} b_{11} 179°, ^{169b} 179–81°, ^{556.5} b_{16} 186°; ¹³⁹⁸ dipole moment 1.97. ^{1113.5}
- p*-Tolylmethyl, (MeC₆H₄CH₂)₂S, m . 76°. ⁸⁵⁰
- 2-*p*-Tolylethyl, (MeC₆H₄CH₂CH₂)₂S, $b_{0.3}$ 60–3°. ⁶⁸²
- 2,4,5-Trimethylphenyl, (Me₃C₆H₂)₂S, m . 111°. ³¹⁷
- 4-Biphenyl, (PhC₆H₄)₂S, m . 172°. ⁴⁷³
- α -Naphthyl, (C₁₀H₇)₂S, m . 110°, ^{555b, 556.5, 724, 727} 106°, ¹¹⁵ 107°, ⁷⁶⁷ 100°; ²⁵ b_{15} 289–90°, ^{724, 727} 290°. ⁷³⁰
- β -Naphthyl, (C₁₀H₇)₂S, m . 151°; ^{555a, 555b, 556.5, 723c, 724, 727, 729} b_{15} 295–6°. ^{727, 730}
- Naphthylmethyl, (C₁₀H₇CH₂)₂S, α m . 119°; β m . 127°. ⁹⁶⁵
- o*-Nitrophenyl, (O₂NC₆H₄)₂S, m . 123°, ^{273, 944} 122°, ⁷⁸⁹ 86°. ⁸⁷⁵
- m*-Nitrophenyl, (O₂NC₆H₄)₂S, m . 193°. ⁹⁰
- p*-Nitrophenyl, (O₂NC₆H₄)₂S, m . 159.5°, ¹⁰⁹³ 158°, ¹²⁵⁷ 157.5°, ⁹⁰⁶ 154°, ^{464, 673, 944, 1337} 143°. ⁸⁷⁵

- 2,4-Dinitrophenyl, $[(\text{O}_2\text{N})_2\text{C}_6\text{H}_3]_2\text{S}$, m. 197° ,^{587, 809} 196° ,¹²⁶³
 194° ,^{159, 248} 193° ,^{309, 497, 498a, 498b} 192° ,^{273, 1414}
- 2,4,6-Trinitrophenyl, $[(\text{O}_2\text{N})_3\text{C}_6\text{H}_2]_2\text{S}$, m. 226° ,¹³⁰⁹ 232° ,^{496.5}
 234° ,^{383.5}
- 2-Nitro-*p*-tolyl, $[\text{Me}(\text{O}_2\text{N})\text{C}_6\text{H}_3]_2\text{S}$, m. 126° .¹⁴⁹
- 3-Nitro-*p*-tolyl, $[\text{Me}(\text{O}_2\text{N})\text{C}_6\text{H}_3]_2\text{S}$, m. $145\text{--}53^\circ$.^{1340.5}
- 4-Nitro-*m*-tolyl, $[\text{Me}(\text{O}_2\text{N})\text{C}_6\text{H}_3]_2\text{S}$, m. 164° .^{587.5}
- 6-Nitro-*o*-tolyl, $[\text{Me}(\text{O}_2\text{N})\text{C}_6\text{H}_3]_2\text{S}$, m. 150° .⁶⁸⁰
- 2-6-Dinitro-*m*-tolyl, $[\text{Me}(\text{O}_2\text{N})_2\text{C}_6\text{H}_2]_2\text{S}$, m. 222° .⁴⁹⁷
- 4,6-Dinitro-*m*-tolyl, $[\text{Me}(\text{O}_2\text{N})_2\text{C}_6\text{H}_2]_2\text{S}$, m. 190° .^{498a}
- o*-Nitrobenzyl, $(\text{O}_2\text{NC}_6\text{H}_4\text{CH}_2)_2\text{S}$, m. 124° .⁶⁵⁴
- m*-Nitrobenzyl, $(\text{O}_2\text{NC}_6\text{H}_4\text{CH}_2)_2\text{S}$, m. 110° .⁸¹²
- p*-Nitrobenzyl, $(\text{O}_2\text{NC}_6\text{H}_4\text{CH}_2)_2\text{S}$, m. 159° .⁶⁰⁶
- 4-Nitro- α -naphthyl, $(\text{NO}_2\text{C}_{10}\text{H}_6)_2\text{S}$, m. 237° .⁸⁰⁸
- 2,4-Dinitro- α -naphthyl, $[(\text{O}_2\text{N})_2\text{C}_{10}\text{H}_5]_2\text{S}$, m. 274° ,¹⁴¹¹ 283° .¹²⁶³
- 3-Phenanthrenyl, m. 225° ; b_2 360° .⁴²⁴
- 5-Nitrothienyl-2, $(\text{O}_2\text{NC}_4\text{H}_2\text{S})_2\text{S}$, m. 106° .³⁴⁰
- 5-Nitrofurfuryl-2, $(\text{O}_2\text{NC}_4\text{H}_2\text{O})_2\text{S}$, m. 99° .^{340, 526, 1178}

Phenyl Aryl Sulfides, PhSar

- o*-Tolyl, $\text{PhSC}_6\text{H}_4\text{Me}$, b_{760} 309.9° ,^{1074a} b_{724} 304.5° ,⁵⁰⁹ b. 300.5° ,¹³⁴⁷
 306.5° , b_{11} 160.5° ,^{169b} 160° ,^{1113.5} b_{50} 200.5° , b_{100} 222.5° ,^{169b} b_7
 150° , b_{49} 200° , b_{92} 220° ; ^{1074a} d 0/4 1.1131, d 15/4 1.1012, d
30/4 1.0893; ^{169b} dipole moment 1.34.^{1113.5}
- m*-Tolyl, $\text{PhSC}_6\text{H}_4\text{Me}$, m. -6.5° ; b_{760} 309.5° ,^{169b, 1074a} b_{11}
 164.5° ,^{169b} 164° ,^{1113.5} b_{50} 203.5° , b_{100} 226° ,^{169b} b_{60} 210° , b_{939}
 320° ; ^{1074a} d 0/4 1.1058, d 15/4 1.0937, d 30/4 1.0816; ^{169b} di-
pole moment 1.62.^{1113.5}
- p*-Tolyl, $\text{PhSC}_6\text{H}_4\text{Me}$, m. 15.7° ; ^{169b} b_{760} 311.5° ,^{169b, 1074a} b_{13}
 169° ,^{1113.5} b_{11} $163\text{--}3.5^\circ$,⁷⁵⁴ 167.5° , b_{50} 206° , b_{100} 228.5° ,^{169b} b_8
 160° , b_{18} 180° , b_{726} 310° , b_{902} 320° ; ^{1074a} d 15.7/4 1.0900; ^{169b}
dipole moment 1.76.^{1113.5}
- Benzyl, PhSCH_2Ph , m. 44.5° ,^{1043b} 44° ,⁵⁷¹ 42° ,^{455b, 1182} 41° ,^{555b,}
^{1261a, 1261d} 40° ; ^{203, 255, 412b} b_4 $122\text{--}4^\circ$,¹⁰⁷⁵ b_{14} $145\text{--}55^\circ$,^{202.5} b_{27}
 197° .¹¹⁸²
- α -Methylbenzyl, PhSCHMePh , m. 161° ; ^{201.3} b_{15} $163\text{--}4^\circ$,³⁶ 167--
 70° ,¹⁹⁹ $b_{1.5}$ $134\text{--}5^\circ$; d 25/4 1.0571; n 25/D 1.5898,⁹⁶⁴ n_D
1.6042.³⁶
- 2-Phenylethyl, $\text{PhSCH}_2\text{CH}_2\text{Ph}$, b_{15} $188\text{--}9^\circ$,³⁶ b_{18} 188° ; ¹⁹⁹ n_D
1.6082.³⁶

- 1-Methyl-2-phenylethyl, $\text{PhSCHMeCH}_2\text{Ph}$, b_{11} 177–8°; d 19/4 1.070; n 25.5/D 1.5982.⁶⁸¹
- Diphenylmethyl, PhSCHPh , m. 82°,⁷⁰⁷ 78.5°,¹²¹ 78°.^{429, 1155}
- Triphenylmethyl, PhSCPh_3 , m. 106.5°,¹¹³ 106°,^{429, 516, 707, 753a} 105°.⁸⁸⁹
- 2,5-Dimethylphenyl, $\text{PhSC}_6\text{H}_3\text{Me}_2$, b_{11} 171°; d 0/4 1.0913, d 15/4 1.0795, d 20/4 1.0677.^{169b}
- 2,4,5-Trimethylphenyl, $\text{PhSC}_6\text{H}_2\text{Me}_3$, b_{11} 180°; d 0/4 1.0817, d 15/4 1.0703.^{169b}
- 1,2-Diphenylethyl, $\text{PhSCHPhCH}_2\text{Ph}$, b_1 175–85°.¹²⁰¹
- β -Styryl, PhSCH:CHPh , b_1 155–60°.¹²⁰¹
- 1-Phenyl-2-methylvinyl, PhSCPh:CHMe , m. 42.5°.^{249.5}
- 1-Phenyl-2,2-dimethylvinyl, PhSCPh:CMe_2 , m. 36°.^{249.5}
- 1,2-Diphenylvinyl, PhSCPh:CHPh , m. 50°; isomer m. 64°.^{249.5}
- 9,10-Diphenylanthracenyl, m. 226°.⁹⁷³
- α -Naphthyl, $\text{PhSC}_{10}\text{H}_7$, m. 41.8°,^{169b} 41.5°,^{555b, 724, 1386} 41°;^{556.5} b_{45} 255–6°,¹³⁸⁶ b_{11} 220.5°,^{169b} 217–22°,^{556.5} b_{14} 218°;⁷²⁴ d 15/4 1.167.¹³⁸⁶
- β -Naphthyl, $\text{PhSC}_{10}\text{H}_7$, m. 51.8°,^{169b} 51.5°,⁷²⁴ 51°;^{555b} b_{14} 222–6°;⁷²⁴ b_{11} 226°.^{169b}

Benzyl Aryl Sulfides, PhCH_2SAr

- Diphenylmethyl, $\text{PhCH}_2\text{SCHPh}_2$, m. 70.5°.¹¹⁵⁵
- α -Phenylethyl, $\text{PhCH}_2\text{SCHMePh}$, $b_{1.5}$ 134–5°; d 25/4 1.0571; n 25/D 1.5898.⁹⁶⁴
- β -Phenylethyl, $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{Ph}$, b_1 154–8°,²²⁰ b_1 190–5°.¹⁰³⁶
- p*-Tolyl, $\text{PhCH}_2\text{SC}_6\text{H}_4\text{Me}$, m. 46°,⁴⁸⁹ 44°.^{455d}
- 2,5-Dimethylphenyl, $\text{PhCH}_2\text{SC}_6\text{H}_4\text{Me}_2$, m. 35°.^{1261d}
- m*-Methylbenzyl, $\text{PhCH}_2\text{SCH}_2\text{C}_6\text{H}_4\text{Me}$, b_{15} 193–6°.¹³⁶²
- 4-Biphenyl, $\text{PhCH}_2\text{SC}_6\text{H}_4\text{Ph}$, m. 128.5°.^{1266b}
- α -Naphthyl, $\text{PhCH}_2\text{SC}_{10}\text{H}_7$, m. 80°.^{1261a, 1261d}
- β -Naphthyl, $\text{PhCH}_2\text{SC}_{10}\text{H}_7$, m. 90.7°,¹⁰⁹² 89°.^{1266b}
- 9,10-Diphenylanthracenyl- α , m. 182°.⁹⁷³

Other Unsymmetrical Aryl Sulfides

- Diphenylmethyl *p*-diphenylmethylphenyl, $\text{Ph}_2\text{CHSC}_6\text{H}_4\text{CHPh}_2$, m. 121°.⁴²⁹
- α -naphthyl, $\text{Ph}_2\text{CHSC}_{10}\text{H}_7$, m. 77.5°,⁴²⁹ 77°,⁷⁰⁷ 78°.¹¹⁵⁵
- β -naphthyl, $\text{Ph}_2\text{CHSC}_{10}\text{H}_7$, m. 123°.⁴²⁹

- 4-diphenylmethyl- α -naphthyl, $\text{Ph}_2\text{CHSC}_{10}\text{H}_6\text{CHPh}_2$, m. 148° .⁴²⁹
- Triphenylmethyl-*o*-tolyl, $\text{Ph}_3\text{CSC}_6\text{H}_4\text{Me}$, m. 145° ,⁴²⁹ 99° .⁵¹⁶
- *m*-tolyl, $\text{Ph}_3\text{CSC}_6\text{H}_4\text{Me}$, m. 92° .⁵¹⁶
- *p*-tolyl, $\text{Ph}_3\text{CSC}_6\text{H}_4\text{Me}$, 149° ,⁶⁹ 147° ,⁴²⁹ 146° .⁵¹⁶
- α -naphthyl, $\text{Ph}_3\text{CSC}_{10}\text{H}_7$, m. 121° .⁴²⁹
- β -naphthyl, $\text{Ph}_3\text{CSC}_{10}\text{H}_7$, m. 134° .⁴²⁹
- α -triphenylmethyl- β -naphthyl, $\text{Ph}_3\text{CSC}_{10}\text{H}_6\text{CPh}_3$, m. 82° .⁴²⁹
- o*-Tolyl *m*-tolyl, $\text{MeC}_6\text{H}_4\text{SC}_6\text{H}_4\text{Me}$, b_{11} 170° .⁸⁶⁷
- *p*-tolyl, $\text{MeC}_6\text{H}_4\text{SC}_6\text{H}_4\text{Me}$, b_{11} 173° ; d 0/4 1.0889, d 15/4 1.0774.^{169b}
- α -styryl, $\text{MeC}_6\text{H}_4\text{SCPh:CH}_2$, b_{12} $183-4^\circ$.^{1122b}
- α -naphthyl, $\text{MeC}_6\text{H}_4\text{SC}_{10}\text{H}_7$, b_{11} 227.5° ; d 15/4 1.1504.^{169b}
- β -naphthyl, $\text{MeC}_6\text{H}_4\text{SC}_{10}\text{H}_7$, b_{12} 232° , d 15/4 1.1420.^{169a}
- m*-Tolyl *p*-tolyl, $\text{MeC}_6\text{H}_4\text{SC}_6\text{H}_4\text{Me}$, m. 27.8° ; b_{11} 179° .^{169b}
- α -naphthyl, $\text{MeC}_6\text{H}_4\text{SC}_{10}\text{H}_7$, b_{12} $229.5-30^\circ$; ^{169a} d 15/4 1.445.^{169a}, ^{169b}
- β -naphthyl, $\text{MeC}_6\text{H}_4\text{SC}_{10}\text{H}_7$, m. 60° ; b_{12} 236° .^{169a}
- p*-Tolyl α -naphthyl, $\text{MeC}_6\text{H}_4\text{SC}_{10}\text{H}_7$, m. 40.5° ; b_{12} $233-4^\circ$.^{169a}
- β -naphthyl, $\text{MeC}_6\text{H}_4\text{SC}_{10}\text{H}_7$, m. 70.5° ; b_{11} $237-8^\circ$.^{169a}
- 2,4-dimethylphenyl, $\text{MeC}_6\text{H}_4\text{SC}_6\text{H}_3\text{Me}_2$, b_{11} 188° ; d 0/4 1.0913, d 15/4 1.0795, d 20/4 1.0677.^{169b}
- 2,5-dimethylphenyl, $\text{MeC}_6\text{H}_4\text{SC}_6\text{H}_3\text{Me}_2$, m. 6° ; b_{11} 185° ; d 0/4 1.0720, d 15/4 1.0606, d 30/4 1.0494.^{169b}
- 2,4,6-trimethylphenyl, $\text{MeC}_6\text{H}_4\text{SC}_6\text{H}_2\text{Me}_3$, m. 89.6° ; b_{11} 190° .^{169b}
- β -2,4,6-trinitrophenylethyl, $\text{MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{C}_6\text{H}_2(\text{NO}_3)_3$, m. 91° .²⁷²
- 2,4-Dimethylphenyl α -styryl, $\text{Me}_2\text{C}_6\text{H}_3\text{SCPh:CH}_2$, b_{14} $197-8^\circ$.^{1122b}
- α -naphthyl, $\text{Me}_2\text{C}_6\text{H}_3\text{SC}_{10}\text{H}_7$, b_{11} 239.5° ; d 15/4 1.1355.^{169b}
- β -naphthyl, $\text{Me}_2\text{C}_6\text{H}_3\text{SC}_{10}\text{H}_7$, m. 39.6° ; b_{11} 243.5° .^{169b}
- 2,5-Dimethylphenyl α -naphthyl, $\text{Me}_2\text{C}_6\text{H}_3\text{SC}_{10}\text{H}_7$, m. 36.2° ; b_{11} 235° .^{169b}
- β -naphthyl, $\text{Me}_2\text{C}_6\text{H}_3\text{SC}_{10}\text{H}_7$, m. 36.7° ; b_{11} 240° .^{169b}
- 3,4-Dimethylphenyl β -naphthyl, $\text{Me}_2\text{C}_6\text{H}_3\text{SC}_{10}\text{H}_7$, m. 68° ; b_{11} 251.5° .^{169b}
- 3,5-Dimethylphenyl α -naphthyl, $\text{Me}_2\text{C}_6\text{H}_3\text{SC}_{10}\text{H}_7$, b_{11} 246° ; d 15/4 1.1346.^{169b}

- 2,4,6-Trimethylphenyl α -naphthyl, $\text{Me}_3\text{C}_6\text{H}_2\text{SC}_{10}\text{H}_7$, m. 120.6° ; b_{11} 245° .^{169b}
 — β -naphthyl, $\text{Me}_3\text{C}_6\text{H}_2\text{SC}_{10}\text{H}_7$, m. 87.5° ; b_{11} 245° .^{169b}
 α -Phenylvinyl β -naphthyl, $\text{CH}_2\text{:CPhSC}_{10}\text{H}_7$, m. 85° ; b_{12} $238-9^\circ$.^{1122c}
 α -Naphthyl β -naphthyl, $\text{C}_{10}\text{H}_7\text{SC}_{10}\text{H}_7$, b_{15} $290-1^\circ$.^{723c}

Nitrophenyl Sulfides

o-Nitrophenyl Sulfides, $o\text{-O}_2\text{NC}_6\text{H}_4\text{SR}$

- Methyl, m. 65° ,^{180, 183 185a} 64° ,^{52, 588, 873} 60° ,^{303, 440} d $78.2/4$ 1.2626; n $78.2/D$ 1.62458.¹⁸³
 Ethyl, b_{15} $149-50^\circ$,⁴⁴⁰ b_{10} 172° ; d_{18} 1.262; n $18/D$ 1.62126.¹⁸⁷
 Propyl, b_7 $172-4^\circ$.⁴⁴⁰
 Allyl, m. 54° .⁴⁴⁰
 Phenyl, m. 81° ,^{753b} 80.2° ,¹⁷² 80.5° ,⁴⁸⁶ 79° ,¹¹⁰⁶ 77° ,⁸⁶⁷ 76° ,^{18, 1038} b_{15} 210° .¹¹⁰⁶
 Benzyl, m. 83° ; b_{12} 230° .¹¹⁸⁸
 o -Tolyl, m. 88° ,⁴⁸⁶ 87° ,⁸⁶⁷ 86° ; b_{18} $210-5^\circ$.¹³¹⁷
 m -Tolyl, m. 86.5° ; ^{486, 1317} b_{18} 222° .¹³¹⁷
 p -Tolyl, m. 90° , ⁴⁸⁶ 87.5° .¹³¹⁷
 2,4-Dinitro- α -naphthyl, m. 226° .^{590a}

m-Nitrophenyl Sulfides, $m\text{-O}_2\text{NC}_6\text{H}_4\text{SR}$

- Methyl, m. 14.5° .¹⁸⁴
 Phenyl, m. 42.5° .^{977a}
 p -Tolyl, m. 61° .^{977a}

p-Nitrophenyl Sulfides, $p\text{-O}_2\text{NC}_6\text{H}_4\text{SR}$

- Methyl, m. 72° ,^{52, 183, 188, 476, 873} 67° ,^{125, 588} d $80.1/4$ 1.2391; n $80.1/D$ 1.64008.¹⁸³
 Ethyl, m. 44° ,^{188 476, 905} 40° .¹²⁵
 Propyl, d $15/4$ 1.1952.¹³³⁷
i-Propyl, m. 44.5° .¹³³⁷
 Butyl, d $25/4$ 1.1591.¹³³⁷
i-Butyl, d $25/4$ 1.1540.¹³³⁷
i-Amyl, d $25/4$ 1.1302.¹³³⁷
 Octyl, m. 35° .^{233a}
 Octadecyl, m. 69° .¹¹⁸⁰
 Cyclohexyl, m. 57° .⁴⁴⁰

Allyl, m. 41° ,⁵⁰ 39° .⁴⁴⁰
 Phenyl, m. 55° ,^{18, 486, 673, 1257} 57° ,¹⁰³⁸ 54.4° ; b_{25} 240° , b_{50} 262.5° ,
 b_{100} 288.2° .¹⁷²
 Benzyl, m. 123° ,¹³³⁷ 122.5° .⁶³
o-Tolyl, m. 65° .⁴⁸⁶
m-Tolyl, m. 47° .⁴⁸⁶
p-Tolyl, m. 82° ,⁷⁰⁹ 81.5° ,⁷⁴⁹ 81° .⁴⁸⁶
i-PrC₆H₄, m. 48.5° .⁴⁸⁶
 α -Naphthyl, m. 87° ,^{49.5} 85° .⁷⁰⁹
o-Nitrophenyl, m. 108.5° .⁸⁷⁵
p-Nitrobenzyl, m. 108° .¹³³⁷
 2,4-Dinitro- α -naphthyl, m. 198° .^{590a}
 4,5-Dinitro- α -naphthyl, m. 221° .^{590a}
 2-Thienyl, m. 62° .^{233b}
 4-Nitro-2-thienyl, m. 133° .³⁴⁰
 4-Nitro-2-furfuryl, m. 119° .³⁴⁰

Other Nitro Sulfides

Methyl *p*-nitro-*o*-tolyl, m. 83° .⁹⁹⁶
 — *o*-nitro-*p*-tolyl, m. 77° .¹⁴¹³
 Ethyl *p*-nitro-*o*-tolyl, m. 78° .⁹⁹⁶
 — *o*-nitro-*p*-tolyl, m. 52° .¹⁴¹³
 Phenyl 6-nitro-*o*-tolyl, m. 82.8° .^{171.5}
 — 2-nitro-*m*-tolyl, m. 72° .^{171.5}
 — 6-nitro-*m*-tolyl, m. 59.5° .^{171.5}
 — 4,6-dinitro-*m*-tolyl, m. 114.5° ,^{171.5} 143° .^{498a}
 Allyl *o*-nitrobenzyl, b_1 138° .⁵⁰
 Methyl *m*-nitrobenzyl, m. 31° .⁸¹²
 Allyl *m*-nitrobenzyl, b_1 138° .⁵⁰
i-Propyl *p*-nitrobenzyl, m. 34° .¹¹⁹
 Butyl *p*-nitrobenzyl, d 25/4 1.1429.¹³³⁷
i-Amyl *p*-nitrobenzyl, d 25/4 1.1480.¹³³⁷
 Dodecyl *p*-nitrobenzyl, m. 33° .¹²³⁰
 Allyl *p*-nitrobenzyl, b_2 $147-8^{\circ}$.⁵⁰
 Phenyl *p*-nitrobenzyl, m. 79° .¹³³⁷
 Benzyl *p*-nitrobenzyl, m. 57° .⁹⁶⁵
 Ethyl 2,4-dinitro- α -naphthyl, m. 85° .¹²⁶³
 2,4,4'-Trinitro-1,1-dinaphthyl, m. 216° .^{590a}

TABLE 14.3
2,4-Dinitrophenyl Sulfides

R	M.p.(°C)	R	M.p.(°C)
Allyl	71.5° ¹⁰⁵ 71° ¹⁰⁴	<i>p</i> -Me ₃ CC ₆ H ₄	131° ²³¹
Propenyl	120° ^{841.5}	2,5-Me ₂ C ₆ H ₃	135° ²³¹
Et ₂ CH	53° ²⁴⁸	2,5-(Me ₂ C) ₂ C ₆ H ₃	151° ²³¹
1-MeC ₅ H ₇	74° ⁸¹³	<i>p</i> -PhCH ₂ CH ₂ C ₆ H ₄	133° ²³¹
C ₆ H ₁₁	147° ²⁴⁸	Ph ₂ C:CH	136.5° ⁶³⁰
	145° ^{1303.5}	Ph ₃ C	190° ⁴²⁰
3-MeC ₆ H ₁₀	143° ^{1303.5}	PhCH ₂ CH ₂	87.5° ^{104, 106}
3-Butenyl	56.5° ^{118b}	PhC ₆ H ₄	146° ¹⁰⁶
C ₁₀ H ₁₈	100° ^{100, 434}	<i>o</i> -O ₂ NC ₆ H ₄	133° ^{800b}
Phenyl	121° ¹⁰⁶ 120.5° ⁴²⁵ 117° ¹⁴⁸	<i>p</i> -O ₂ NC ₆ H ₄	131° ¹²⁶ 160° ⁹⁶⁵ 155° ¹²⁶
Benzyl	130° ^{68, 104, 106} 128° ^{1369a}	<i>o</i> -MeC ₆ H ₄ CH ₂	168° ⁵⁵⁷
<i>o</i> -Tolyl	101° ¹⁶⁵	<i>o</i> -MeC ₆ H ₄ CHPh	123° ⁵⁵⁷
<i>m</i> -Tolyl	100.5° ⁴⁸⁵ 90.5° ¹⁰⁵	α -Naphthyl	176° ¹⁶⁵
<i>p</i> -Tolyl	103.5° ⁴²⁵ 103° ¹⁰⁶	β -Naphthyl	145° ¹⁶⁵
<i>p</i> -EtC ₆ H ₄	97.5° ²³¹	4-O ₂ N- α -naphthyl	193° ^{590a}
<i>p</i> -Me ₂ CHC ₆ H ₄	96.5° ⁴⁹⁶	2-Thienyl	149° ²⁴³ 119° ¹⁰⁶
<i>p</i> -BuC ₆ H ₄	73.5° ²³¹	3-Thienyl	133.5° ²⁴³ 133° ²⁰⁰
<i>p</i> -MeEtCHC ₆ H ₄	89° ²³¹	3-Furfurylmethyl	146° ¹⁰⁶
<i>p</i> -Me ₂ CHCH ₂ C ₆ H ₄	100° ²³¹	2,3,5,4,6-Me ₅ (O ₂ N) ₂ C ₆	215° ⁶¹²

The melting points of a number of aliphatic 2,4-dinitrophenyl sulfides have been given in Chapter 1, Volume I, under identification of mercaptans.

TABLE 15.3
2,4,6-Trinitrophenyl Sulfides

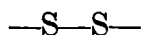
R	M.p.(°C)	R	M.p.(°C)
Methyl	98° ¹²⁶	<i>o</i> -Tolyl	148° ^{977a}
Phenyl	120° ^{977a}	<i>m</i> -Tolyl	102° ^{977a}
<i>p</i> -Ethylphenyl	116° ¹⁰⁰⁹	<i>p</i> -Tolyl	154° ^{977a}
2,4-Dinitrophenyl	217° ^{1859b}		

*Thiophene Sulfides*Thienyl, $C_4H_3S \cdot SR$

- 2-Thienyl methyl, b. 186° ,⁸⁹⁴ $181.5-3.5^\circ$; ¹²²⁵ $5HgCl_2$ m. 168° .¹²²⁵
 — ethyl, b. $196-7^\circ$; $5HgCl_2$, m. 140° .¹²²⁵
 3-Thienyl *i*-propyl, b_3 $65-6^\circ$; ^{209, 210a} $d^{20}/4$ 1.0742; n^{25}/D 1.5532.²⁰⁹
 — *t*-butyl, b_1 $65-7^\circ$; n^{25}/D 1.5440.²⁰⁹
 — octyl, b_1 $108-11^\circ$.²⁰⁹
 — 1-methylheptyl, b_1 $108-11^\circ$.^{210a}
 — 1,1,4,4-tetramethylbutyl, $b_{1.5}$ $102-10^\circ$,^{210a} b_1 $102-13^\circ$; ²⁰⁹ $d^{20}/4$ 1.0140; n^{25}/D 1.5356.^{209, 210a}
 — nonyl, b_1 $105-10^\circ$; $d^{25}/4$ 1.0261; n^{25}/D 1.5403.^{210a}
 — *t*-dodecyl, b_8 $174-6^\circ$; n^{25}/D 1.5343.²⁰⁹
 — tetradecyl, m. 45° .^{209, 210a}
 — allyl, $b_{0.4}$ $50-4^\circ$; ^{209, 210a, 210b} $d^{20}/4$ 1.1541; n^{25}/D 1.5964.²⁰⁹
 — benzyl, m. 38° .^{209, 210a}
 — 2-phenylethyl, $b_{0.5}$ $120-2^\circ$; $d^{20}/4$ 1.1591; n^{25}/D 1.6168.²⁰⁹
 Di-2-thienyl sulfide, b_8 $130-5^\circ$.^{715.5}

Thenyl, $C_4H_3S \cdot CH_2SR$

- 2-Thenyl methyl, b_{15} 94.5° ; d 20.5/4 1.146; n^{20}/D 1.5775.^{244b}
 — ethyl, b_3 $66-9^\circ$.^{693a}
 — *i*-butyl, $b_{3.5}$ $89-91^\circ$.^{693a}
 — hexyl, b_2 $106-9^\circ$.^{693a}
 — benzyl, $b_{1.5}$ $120-1^\circ$.^{693a}
 Di-2-thenyl, $b_{1.5}$ $129-31^\circ$.^{693a}

Multiple Sulfides $RS(CH_2)_nSR$

- $MeSCH_2SMe$, b. $148-9^\circ$,¹⁴¹ 147° ,¹¹⁴⁷ 148° .^{138, 412c}
 $EtSCH_2SEt$, b. 184° ,^{135a, 353, 700a, 770} $180-5^\circ$,⁴⁰⁵ $180-2^\circ$,¹³⁷ $178-81^\circ$; ^{455a} $d^{20}/4$ 0.985,^{353, 700a} 0.987.³⁵³
 $PrSCH_2SPr$, b. 216° ,⁷⁷⁰ b_{13} $94-5^\circ$; ^{55a} $d^{14}/4$ 0.966.⁷⁷⁰
 $BuSCH_2SBu$, b. 250° ,¹²⁴⁰ b_{43} 146° ,^{1366b} b_{28} 144° ; ^{412c} $d^0/4$ 0.9479, $d^{25}/4$ 0.9305,^{1366b} $d^{20}/4$ 0.945; ¹²⁴⁰ n^{20}/D 1.4964,^{1366b} 1.4946.¹²⁴⁰
 $Me_3CSCH_2SCMe_3$, m. -4° ; b_{13} $99-101^\circ$,^{55a} b_5 $72-6^\circ$.¹²⁰⁵
 $H_2C(SCH_2CH_2CHMe_2)_2$, b. 265° ; $d^{14}/4$ 0.924.⁷⁷⁰
 $C_{12}H_{25}SCH_2SC_{12}H_{25}$, m. 42.5° .¹²³⁰

- MeSCH_2SEt , b. $163-7^\circ$,⁴⁸² $164-7^\circ$.¹⁴¹
 EtSCH_2SPr , b_{12} $87-9^\circ$.¹⁴¹
 MeSCH_2SPh , b_{11} $148-52^\circ$.⁴⁸²
 EtSCH_2SPh , b_{12} 145° ,¹⁴¹ $147-51^\circ$; n^{18}/D 1.598.⁴⁸²
 $\text{EtSCH}_2\text{SCH}_2\text{Ph}$, b_{12} 155° , b_{19} 165° .¹³⁷
 PhSCH_2SPh , m. 52° ,^{555a} 36° ,¹¹⁸⁴ 40° .⁹⁶²
 $\text{PhCH}_2\text{SCH}_2\text{SCH}_2\text{Ph}$, m. 55° .^{137, 458, 1360}
p- $\text{MeC}_6\text{H}_4\text{SCH}_2\text{SC}_6\text{H}_4\text{Me}$ -*p*, m. 30° .^{555a}
o- $\text{O}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{SC}_6\text{H}_4\text{NO}_2$ -*o*, m. 170° .⁴⁴⁰
p- $\text{O}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{SC}_6\text{H}_4\text{NO}_2$ -*p*, m. 179° .¹³³⁷
2,4-(O_2N) $_2\text{C}_6\text{H}_3\text{SCH}_2\text{SC}_6\text{H}_3(\text{NO}_2)_2$ -2,4, m. 207° .¹⁶⁴
 $\beta\text{-C}_{10}\text{H}_7\text{SCH}_2\text{SC}_{10}\text{H}_7\text{-}\beta$, m. 104° .^{555a}
 $\text{MeSCH}_2\text{CH}_2\text{SMe}$, b. 183° ; $^{127a, 306, 353, 403}$ $b_{20.5}$ 80° ; 863a $d_{15/4}$ 1.04189, $d_{20/4}$ 1.0366,³⁰⁶ 10371 , $d_{25/4}$ 1.0370; n^{20}/D 1.5296,^{863a} n^{20}/α 1.53026.³⁰⁶
 $\text{EtSCH}_2\text{CH}_2\text{SEt}$, b. 217° ,^{825a} $210-2^\circ$,^{893b} $210-3^\circ$,^{127a, 403, 863a} $207-10^\circ$,¹³⁴⁵ b_{40} 120° ,⁶²⁷ b_{13} 95° ,^{863a} b_{24} $90-5^\circ$,^{632a} $b_{4.5}$ $69.5-70^\circ$,^{1035.5} b_4 64° ,⁷⁵⁰ b_{27} $108-9^\circ$; 546 d_{15} 0.987,³⁵³ $d_{15.5}$ 0.98705,^{893b} d_{20} 0.977,^{825a} d 20/4 0.9815; 863a n 20/D 1.5101,^{825a} n 25/D 1.5095.^{863a}
 $\text{Me}_2\text{CHSCH}_2\text{CH}_2\text{SCHMe}_2$, b_4 $67-70^\circ$.¹⁰⁷⁵
 $\text{BuSCH}_2\text{CH}_2\text{SBu}$, b_3 $106-7^\circ$,^{1366a} b_5 $129-30^\circ$; d 0/4 0.9521, d 25/4 0.9361; n 20/D 1.4962.^{1366b}
 $\text{Me}_3\text{CSCH}_2\text{CH}_2\text{SCMe}_3$, b_{29} $127-9^\circ$.¹²⁰⁵
i- $\text{AmSCH}_2\text{CH}_2\text{SAm}$ -*i*, b. $245-55^\circ$,^{127a, 403} b_4 $127-8^\circ$; d 20/4 0.9214; n 20/D 1.4900.⁶⁸⁹
 $\text{C}_{18}\text{H}_{37}\text{SCH}_2\text{CH}_2\text{SC}_{18}\text{H}_{37}$, m. 72° .¹¹⁸⁰
 $\text{C}_6\text{H}_{11}\text{SCH}_2\text{CH}_2\text{SC}_6\text{H}_{11}$, $b_{0.2}$ $135-7^\circ$.^{1349b}
 $\text{H}_2\text{C}:\text{CHCH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}:\text{CH}_2$, b_7 109° ; d_{25} 0.9998; n 25/D 1.5345.²³⁶
 $\text{MeSCH}_2\text{CH}_2\text{SPh}$, b_3 130° .²⁸¹
 $\text{PhSCH}_2\text{CH}_2\text{SPh}$, m. 65° ,^{127a, 403} 70° ,^{632a, 637a} 69° .^{91, 582.5, 1093}
 $\text{PhSCH}_2\text{CH}_2\text{SC}_6\text{H}_4\text{NO}_2$ -*p*, m. 80° .^{101a}
o- $\text{O}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{SC}_6\text{H}_4\text{NO}_2$ -*o*, m. 207° ,⁴⁵⁷ 208° ,³⁰³ 205° .⁴⁴⁰
p- $\text{O}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{SC}_6\text{H}_4\text{NO}_2$ -*p*, m. 134° ,⁴⁵⁷ 136° .¹³³⁷
2,4-(O_2N) $_2\text{C}_6\text{H}_3\text{SCH}_2\text{CH}_2\text{SC}_6\text{H}_3(\text{NO}_2)_2$ -2,4, m. 248° ,^{164, 165} 250° .^{517.5}
 $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{Ph}$, m. 38° ,⁴⁵⁷ 40.4° .^{863a}
p- $\text{MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{SC}_6\text{H}_4\text{Me}$ -*p*, m. 92° ,^{1203.5} 81° ,^{91, 632a, 637c} 80° ; $^{377, 462}$ b_{3-4} $185-96^\circ$,^{632a} b_2 114° .^{637c}

- $3\text{-C}_4\text{H}_2\text{S}\cdot\text{SCH}_2\text{CH}_2\text{S}\cdot\text{C}_4\text{H}_2\text{S-3}$, m. 55° .^{209, 210a}
 $\text{MeSCH}_2\text{CH}_2\text{SCH}:\text{CH}_2$, b_2 65° .²²¹
 $\text{EtSCH}_2\text{CH}_2\text{SCH}:\text{CH}_2$, b. $200\text{--}14^\circ$,^{893b} $210\text{--}5^\circ$; d 7.5/4 1.0253, d 15/4 1.0188.³⁵⁵
 $p\text{-MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{SC}_{10}\text{H}_7\text{-}\beta$, m. 76° .^{637c}
 $\text{EtSCH}_2\text{CH}_2\text{SO}_2\text{C}_6\text{H}_4\text{Me}$, m. 85° .^{631c, 638}
 $\text{MeSCHMeCH}_2\text{SO}_2\text{CH}_2\text{Ph}$, m. 51° ; disulfone m. 115° .^{860.5}
 $\text{PhCH}_2\text{SCHMeCH}_2\text{SCH}_2\text{Ph}$, b_{14} $218\text{--}22^\circ$.^{1119a}
 $\text{MeSCHPhCH}_2\text{SMe}$, b_{10} $149\text{--}50^\circ$; d 20/4 1.096.^{602c}
 $\text{EtSCHPhCH}_2\text{SPh}$, b_{11} $163\text{--}4^\circ$; d 20/4 1.046.^{602c}
 $\text{PhSCHPhCH}_2\text{SPh}$, m. 58° ,^{602c} b_1 $175\text{--}85^\circ$.¹²⁰¹
 $\text{MeSCH}_2\text{CH}_2\text{CH}_2\text{SMe}$, b_{12} 86.5° ; d 20/4 1.0115, d 23.4/4 1.0085; n 20/D 1.5186, n 25/D 1.5165.^{863a}
 $\text{EtSCH}_2\text{CH}_2\text{CH}_2\text{SEt}$, b. $228\text{--}31^\circ$,^{863a, 1345} b_{10} 107° ; d 20/4 0.9664; n 20/D 1.5052.^{863a}
 $\text{PrSCH}_2\text{CH}_2\text{CH}_2\text{SBu}$, b_{15} $145\text{--}8^\circ$.^{152.5}
 $\text{Me}_3\text{CSCH}_2\text{CH}_2\text{CH}_2\text{SCMe}_3$, $b_{4.5}$ 120° .⁴¹¹
 $\text{Me}_2\text{CHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CHMe}_2$, b_5 $147\text{--}8^\circ$.⁴¹¹
 $\text{C}_6\text{H}_{11}\text{SCH}_2\text{CH}_2\text{CH}_2\text{SC}_6\text{H}_{11}$, $b_{0.1}$ $141\text{--}3^\circ$.^{336d}
 $\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{SPh}$, b_{12} 264° .⁸⁴¹
 $o\text{-NO}_2\text{C}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{CH}_2\text{SC}_6\text{H}_4\text{NO}_2\text{-}o$, m. 140° .⁴⁴⁰
 $p\text{-O}_2\text{NC}_6\text{H}_4\text{S}(\text{CH}_2)_3\text{SC}_6\text{H}_4\text{NO}_2\text{-}p$, m. 110° .¹³³⁷
 $2,4\text{-(O}_2\text{N)}_2\text{C}_6\text{H}_3\text{S}(\text{CH}_2)_3\text{SC}_6\text{H}_3(\text{NO}_2)_2\text{-}2,4$, m. 194° ,¹⁶⁵ 200° .^{517.5}
 $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{Ph}$, m. 6° ; b_5 $219.5\text{--}2^\circ$,^{863a} b_8 $218\text{--}21^\circ$.⁴²
 $\text{EtSCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{Ph}$, $b_{0.3}$ 135° .^{1119b}
 $\text{EtSCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{SEt}$, b_{24} $90\text{--}5^\circ$.^{637a}
 $\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{SPh}$, m. 84.5° ,⁸⁵⁵ 85° ,¹⁰⁰ 89.1° .¹⁷
 $2,4\text{-(O}_2\text{N)}_2\text{C}_6\text{H}_3\text{S}(\text{CH}_2)_4\text{SC}_6\text{H}_3(\text{NO}_2)_2\text{-}2,4$, m. 176° ,¹⁶⁵ 230° .^{517.5}
 $p\text{-MeC}_6\text{H}_4\text{S}(\text{CH}_2)_4\text{SC}_6\text{H}_4\text{Me-}p$, m. 65.5° .⁸⁵⁵
 $2\text{-C}_4\text{H}_3\text{S}\cdot\text{S}(\text{CH}_2)_4\text{SC}_4\text{H}_3\text{S-}2$, m. 63° .^{209, 210a}
 $\text{PhSCMe}_2\text{CH}_2\text{CH}_2\text{CMe}_2\text{SPh}$, m. 80° .²²⁷
 $o\text{-MeC}_6\text{H}_4\text{SCMe}_2\text{CH}_2\text{CH}_2\text{CMe}_2\text{SC}_6\text{H}_4\text{Me-}o$, m. 76° .²²⁷
 $m\text{-MeC}_6\text{H}_4\text{SCMe}_2\text{CH}_2\text{CH}_2\text{CMe}_2\text{SC}_6\text{H}_4\text{Me-}m$, m. 106° .²²⁷
 $p\text{-MeC}_6\text{H}_4\text{SCMe}_2\text{CH}_2\text{CH}_2\text{CMe}_2\text{SC}_6\text{H}_4\text{Me-}p$, m. 129° .²²⁷
 $2,4\text{-(O}_2\text{N)}_2\text{C}_6\text{H}_3\text{S}(\text{CH}_2)_5\text{SC}_6\text{H}_3(\text{NO}_2)_2\text{-}2,4$, m. 170° ,¹⁶⁵ 176° .^{517.5}
 $\text{C}_6\text{H}_{11}\text{S}(\text{CH}_2)_6\text{SC}_6\text{H}_{11}$, $b_{0.1}$ 166° .^{336d}
 $\text{PhS}(\text{CH}_2)_6\text{SPh}$, m. 82° .^{152.5}
 $2,4\text{-(O}_2\text{N)}_2\text{C}_6\text{H}_3\text{S}(\text{CH}_2)_6\text{SC}_6\text{H}_3(\text{NO}_2)_2\text{-}2,4$, m. 218° ,¹⁶⁵ 222° .^{517.5}
 $(\text{MeCH}:\text{CHCH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\cdot)_2$, $b_{0.5}$ $35\text{--}40^\circ$; n 20/D 1.5150.⁸⁵⁶

2,4-(O₂N)₂C₆H₃S(CH₂)₇SC₆H₃(NO₂)₂-2,4, m. 139°. ^{517.5}

PhS(CH₂)₈SPh, m. 83°. ^{190a}

2,4-(O₂N)₂C₆H₃S(CH₂)₈SC₆H₃(NO₂)₂-2,4, m. 187°. ^{517.5}

2,4-(O₂N)₂C₆H₃S(CH₂)₉SC₆H₃(NO₂)₂-2,4, m. 132°. ^{517.5}

PhS(CH₂)₁₀SPh, m. 85°. ^{190a}

2,4-(O₂N)₂C₆H₃S(CH₂)₁₀SC₆H₃(NO₂)₂-2,4, m. 175°. ^{517.5}

2,4-(O₂N)₂C₆H₃S(CH₂)₁₂SC₆H₃(NO₂)₂-2,4, m. 174°. ^{517.5}

For a large number of compounds of this class see Table 9.3.

RSCH:CHSR

EtSCH:CHSEt, b₁₃ 170°. ⁴⁵⁷

EtSCH:CMeSEt, b₉ 95–7°. ^{1119c}

PhSCH:CHSPh, m. 62°; b. 235–42°; ³³⁸ *cis* m. 32.5°; *trans* b_{0.24} 150–5°. ^{973.5}

PhCH₂SCH:CHSCH₂Ph, m. 61°. ⁴⁵⁷

p-MeC₆H₄SCH:CHSC₆H₄Me-*p*, m. 93°, ⁴⁶³ 91°. ^{1303.5}

o-O₂NC₆H₄SCH:CHSC₆H₄NO₂-*o*, m. 215°. ⁴⁵⁷

p-O₂NC₆H₄SCH:CHSC₆H₄NO₂-*p*, m. 126°. ⁴⁵⁷

RSC:CSR

PhCH₂SC:CSCH₂Ph, m. 53°. ⁴⁵⁷

p-MeC₆H₄SC:CSCH₂Ph, m. 100°. ⁴⁶³

o-O₂NC₆H₄SC:CSCH₂Ph, m. 225°. ⁴⁵⁷

RSC₆H₄SR

MeSC₆H₄SMe, *meta* b₁₇ 149°; ¹⁴¹² *para* m. 85°. ^{1407a}

p-MeSO₂C₆H₄SMe, m. 99°. ²³⁴

EtSC₆H₄SEt, *meta* b₁₈₋₉ 164°; ¹⁴¹² *para* m. 46.5°. ^{101a}

p-PhSC₆H₄SPh, m. 81.5°, ¹⁷¹ 80°; ^{584d} b₁₄ 265°. ¹⁷¹

PhCH₂SC₆H₄SCH₂Ph, *meta* m. 60°, ⁴³⁰ 50°; ¹⁴¹² *para* m. 127°. ⁴³⁰

p-(*p*-MeC₆H₄S)₂C₆H₄, m. 99°; b₁₄ 284°. ¹⁷¹

p-PhSC₆H₄SC₆H₄Me-*p*, m. 55.5°; b₁₄ 272°. ¹⁷¹

p-(α-C₁₀H₇S)₂C₆H₄, m. 148.5°; b₁₄ 253°. ¹⁷¹

p-(β-C₁₀H₇S)₂C₆H₄, m. 185°. ¹⁷¹

o-[2,4,6-(O₂N)₃C₆H₂S]₂C₆H₄, m. 269°. ^{1007a}

1,3-(MeS)₂C₆H₃NO₂-4, m. 114°. ¹⁴¹²

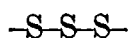
2,4-(MeS)₂C₆H₃Et, b₁₄ 171–3°. ¹⁰⁰⁹

1,3-(MeS)₂C₆H₂(NO₂)Et-4,5, m. 118°. ¹⁰⁰⁹

1,3-(MeS)₂C₆H₂Me₂-4,5, m. 39°. ¹⁰¹⁰

1,3-(MeS)₂C₆H₂Me₂-2,4, m. 84.5°. ¹⁰¹¹

- 1,3-(MeS)₂C₆H₂Me₂-2,5, m. 94°. ¹⁰¹¹
 1,4-(MeS)₂C₆H₂Me₂-3,5, b₁₄ 167-9°. ¹⁰¹¹
 1,3-[2,4,6-(O₂N)₃C₆H₂S]₂C₆H₂Me₂-2,4, m. 259.5°. ¹⁰¹¹
 1,3-[2,4,6-(O₂N)₃C₆H₂S]₂C₆H₂Me₂-2,5, m. 251-5°. ¹⁰¹¹
 1,4-[2,4,6-(O₂N)₃C₆H₂S]₂C₆H₂Me₂-2,6, m. 213°. ¹⁰¹¹
 2,4-[2,4,6-(O₂N)₃C₆H₂S]₂C₆H₃Et, m. 199°. ¹⁰⁰⁹
 1,3-(PhCH₂S)₂C₆H₃NO₂-4, m. 92°. ⁴³⁰
p-MeSC₆H₄C₆H₄SMe-*p*, m. 185°, ¹⁴⁰⁵ 184°, ⁷⁶⁷ 186°. ⁹⁵⁰
p-EtSC₆H₄C₆H₄SEt-*p*, m. 135°. ^{767, 1405}
p-PhCH₂SC₆H₄C₆H₄SCH₂Ph, m. 199°. ¹⁴⁰⁵
p-MeSC₆H₄CH₂CH₂CH₂C₆H₄SMe-*p*, m. 156°. ²³⁸
p-MeSC₆H₄CH:CHC₆H₄SMe-*p*, m. 208.5°. ^{241a, 1324}
 (*p*-MeSC₆H₄)₂C:CHPh, m. 106°. ⁹²⁶
 2,6-(*p*-MeC₆H₄S)₂C₆H₃NO₂, m. 168°. ⁸⁰¹
 (*p*-EtSC₆H₄)₂C:CH₂, m. 118°. ^{1261.5}



- EtSCH₂CH₂SEt, b₁₇ 135°. ¹³⁷
 MeSCH₂CH₂SCH₂CH₂SMe, m. 24.32°, ¹¹¹⁸ 24.3°, ⁸⁷⁶ 27-8°; ^{863a} b. 260°, b₁ 118°, b_{2.5} 125°, b₅ 137°, ⁸⁷⁶ b₁₀ 146°, ²² b₈ 145°; d 20/4 1.1101, d 28.5/4 1.1052, ^{863a} d 25/4 1.08935; n 20/D 1.55835, ⁸⁷⁶ 1.5672, n 24/D 1.5653. ^{863a}
 MeSCH₂CH₂SO₂CH₂CH₂SMe, m. 77°. ⁵⁶³
 MeSO₂CH₂CH₂SCH₂CH₂SO₂Me, m. 116°. ⁶³⁸
 MeSCH₂CH(SMe)CH₂SMe, b₁₅ 147°. ¹⁰⁹⁶
 (EtSCH₂CH₂)₂S, m. 17°, ⁵⁶³ 10°; ^{863a} b. 173-5°, ⁵⁶³ b₈₋₉ 157°; d 25/4 1.0596; n 25/D 1.5489, ^{863a} n 22.5/D 1.5150. ⁵⁶³
 (EtSCH₂CH₂)₂SO₂, m. 64°. ⁵⁶³
 (PrSCH₂CH₂)₂S, m. 27.5°; b. 193-5°. ⁵⁶³
 (PrSCH₂CH₂)₂SO₂, m. 75.5°. ⁵⁶³
 (BuSCH₂CH₂)₂S, m. 17.5°; b. 222-3°; d₂₀ 1.102; n 22.5/D 1.5455. ⁵⁶³
 (BuSCH₂CH₂)₂SO, m. 25°; (BuSCH₂CH₂)₂SO₂, m. 73.7°. ⁵⁶³
 (*i*-BuSCH₂CH₂)₂SO₂, m. 94.2°. ⁵⁶³
 (*s*-BuSCH₂CH₂)₂SO₂, m. 15°. ⁵⁶³
 (AmSCH₂CH₂)₂S, b₂ 190-210°. ⁷⁶
 (*i*-AmSCH₂CH₂)₂S, m. 20°; b. 170°; (*i*-AmSCH₂CH₂)₂SO₂, m. 91°. ⁵⁶³
 (2-C₄H₃S·SCH₂CH₂)₂S, m. 56°. ^{1075.5}

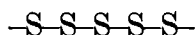
For more compounds of this class see Table 6.3.

- $(\text{PhSCH}_2\text{CH}_2)_2\text{S}$, m. 57.5° ,⁵⁶³ 57° ; ¹⁰ $(\text{PhSCH}_2\text{CH}_2)_2\text{SO}$, m. 121° ; ⁵⁶³ $(\text{PhSCH}_2\text{CH}_2)_2\text{SO}_2$ m. 104.5° .^{735b}
 $(\text{PhSO}_2\text{CH}_2\text{CH}_2)_2\text{S}$, m. 124° .^{961c}
 $(p\text{-MeC}_6\text{H}_4\text{SO}_2\text{CH}_2\text{CH}_2)_2\text{S}$, m. 16° .⁶³⁸
 $(\text{O}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{CH}_2)_2\text{S}$, *ortho* m. 108° ; ⁴⁴⁰ *para* m. 86.5° .¹³³⁷
 $[2,4\text{-(O}_2\text{N)}_2\text{C}_6\text{H}_3\text{SCH}_2\text{CH}_2]_2\text{S}$, m. 160° .^{517.5}
 $(\text{PhCH}_2\text{SCH}_2\text{CH}_2)_2\text{S}$, m. 53° ,⁴⁵⁹ 39° ,²² 38° .^{863a}
 $(\text{PhSCH}:\text{CH})_2\text{S}$, m. 78° .³³⁸
 $p\text{-MeSC}_6\text{H}_4\text{SC}_6\text{H}_4\text{SMe-}p$, m. 89° .¹²⁹⁹
 $p\text{-MeSO}_2\text{C}_6\text{H}_4\text{SC}_6\text{H}_4\text{SO}_2\text{Me-}p$, m. 197° .²³⁴
 $p\text{-EtSC}_6\text{H}_4\text{SC}_6\text{H}_4\text{SEt-}p$, m. 38.5° .¹²⁹⁹
 $\text{PhSCH}_2\text{CH}(\text{CH}_2\text{SO}_2\text{Ph})\text{SPh}$, m. $75\text{--}7^\circ$.^{961a}
 $\text{MeC}(\text{CH}_2\text{SEt})_3$, b_2 $140\text{--}1^\circ$.⁴¹¹
 $\text{MeC}(\text{CH}_2\text{SPr})_3$, $b_{3.5}$ $173\text{--}5^\circ$.⁴¹¹
 $\text{MeC}(\text{CH}_2\text{SBu})_3$, b_3 $205\text{--}6^\circ$.⁴¹¹
 $\text{MeC}(\text{CH}_2\text{SCH}_2\text{CH}_2\text{CHMe}_2)_3$, b_2 $202\text{--}3^\circ$.⁴¹¹
 $\text{HC}(\text{C}_6\text{H}_4\text{SMe-}p)_3$, m. 95.5° .^{185b}
 $\text{HC}(\text{C}_6\text{H}_4\text{SPh})_3$, m. 109° .³⁶⁵
 $1,2,4\text{-(MeS)}_3\text{C}_6\text{H}_3$, m. $49\text{--}53^\circ$.^{1007b}
 $\text{EtSCHMeCH}(\text{SEt})_2$, $b_{0.3}$ 100° .^{1119c}
 $\text{EtSCH}_2\text{CH}_2\text{CH}(\text{SEt})_2$, $b_{0.2}$ 87° , b_9 $94\text{--}7^\circ$.^{1119c}
 $\text{EtSCMe:CHCH}(\text{SEt})_2$, b_9 $210\text{--}3^\circ$.³⁷⁶
 $\text{EtSPh:CHCH}(\text{SEt})_2$, b_{10} $198\text{--}203^\circ$.³⁷⁶



- $(\text{--CH}_2\text{SCH}_2\text{CH}_2\text{SMe})_2$, m. 66.5° ,⁸⁷⁶ 1118 64° ; ⁴⁶⁹ b_8 $180\text{--}5^\circ$.⁸⁷⁶
 $\text{C}(\text{SMe})_4$, m. 65° ,^{55b} 65.5° ; ⁵⁴ b_{12} 126.7° ; ^{55b} transition temperatures 23.2° and 45.5° ; d 20/4 1.321, d 24/4 1.267, d 43/4 1.261, d 70/4 1.174; dielectric constant 2.344 at 20° , 2.818 at 70° ; dipole moment 0.50; surface tension 36 dynes/cm. at 70° ; parachor 419; heats of transition 1460 and 1820 cal/cc.⁵⁴, ^{988b}
 $\text{C}(\text{SEt})_4$, m. 33.5° .^{55b}
 $\text{C}(\text{SCHMe}_2)_4$, m. 61.4° .^{55b}
 $\text{C}(\text{SPh})_4$, m. 159° .^{55c}
 $\text{C}(\text{SC}_8\text{H}_{11})_4$, m. 169° .^{55c}
 $\text{C}(\text{SC}_{10}\text{H}_7\text{--}\beta)_4$, m. 136° .^{55c}
 $(\text{EtS})_2\text{C}(\text{CH}_2\text{SEt})_2$, b_{15} $51\text{--}3^\circ$.¹¹¹³
 $\text{C}(\text{CH}_2\text{SMe})_4$, b_{15} $209\text{--}11^\circ$; d 15/4 1.144; n 15/D 1.572.^{51a}
 $\text{C}(\text{CH}_2\text{SEt})_4$, b_5 $190\text{--}2^\circ$; b_{14} $216\text{--}8^\circ$,²⁹³ b_{15} $218\text{--}20^\circ$; d 15/4 1.066; n 15/D 1.545.^{51a}

- $C(CH_2SPr)_4$, b_3 222–5°; d 15/4 1.029; n 15/D 1.530.^{51a}
 $C(CH_2SCHMe_2)_4$, b_2 192–3°; d 15/4 1.007; n 15/D 1.524.^{51a}
 $C(CH_2SBu)_4$, b_2 225–9°,⁴¹¹ 226–30°; d 15/4 1.001; n 15/D 1.520.^{51a}
 $C(CH_2SCHMeEt)_4$, b_2 221–3°; d 15/4 0.991; n 15/D 1.521.⁵⁷
 $C(CH_2SCH_2CHMe_2)_4$, b_2 206–8°; d 15/4 0.988; n 15/D 1.516.^{51a}
 $C(CH_2SCMe_3)_4$, m . 123.5°,^{51a} 116.4°.⁵⁷
 $C(CH_2SAm)_4$, b_2 241–3°; d 15/4 0.9800; n 15/D 1.514.⁵⁷
 $C(CH_2SAm-i)_4$, b_2 220–2°.⁴¹¹
 $C(CH_2SHex)_4$, d 15/4 0.9630; n 15/D 1.508.⁵⁷
 $C(CH_2SHep)_4$, d 15/4 0.9495; n 15/D 1.504.⁵⁷
 $C(CH_2SOct)_4$, d 15/4 0.9436; n 15/D 1.501.⁵⁷
 $C(CH_2SCH_2CHEtBu)_4$, b 107–16°; d 20/4 0.9475; n 20/D 1.5042.¹¹³⁵
 $C(CH_2SNon)_4$, d 15/4 0.9396; n 15/D 1.499.⁵⁷
 $C(CH_2SDec)_4$, m . 19°.⁵⁷
 $C(CH_2SUnd)_4$, m . 28°.⁵⁷
 $C(CH_2SDod)_4$, m . 36°.⁵⁷
 $(DodSCH_2)_2C(CH_2SCHEtBu)_2$, d 20/4 0.9337; n 20/D 1.4984.¹¹³⁵
 $C(CH_2SCH_2CH:CH_2)_4$, b_2 214–7°; d 15/4 1.084; n 15/D 1.583.^{51a}
 $C(CH_2SPh)_4$, m . 80°.^{51a}
 $C(CH_2SCH_2Ph)_4$, m . 73°.^{51a}
 $(PhCH_2S)_2CHCH(SCH_2Ph)_2$, m . 63°.⁴⁶⁰
1,2,4,5-(MeS)₄C₆H₂, m . 123–7°.^{1009b}



- $(MeSCH_2SCH_2)_2S$, m . 93°.^{584c}
 $(MeSCH_2CH_2SCH_2CH_2)_2S$, m . 88.0°,⁸⁷⁶ 1118 83°,⁴⁶⁹ 84°.²²¹

Symmetrical Selenides, $RSeR$

- Methyl, Me_2Se , b . 58.2°.⁶⁵³
Ethyl, Et_2Se , b . 108°,¹⁰⁰² 107–8°; ¹⁰⁵⁵ d 20/4 1.2300; surface tension 22.94 dynes/cm.; parachor 256.1.⁷⁸
Propyl, Pr_2Se , b . 159°; d 24.3/4 1.1427; n_D 1.4749.^{288b}
Decyl, Dec_2Se , m . 12°; b_3 182–5°.^{356b}
Dodecyl, Dod_2Se , m . 25°; b_3 201–9°.^{356b}
Cetyl, Cet_2Se , m . 125°.³⁵⁷
Phenyl, Ph_2Se , m . 2.5°; ^{974b} b . 302°,⁷²⁵ 726b 303°,⁴³⁹ 301–2°,⁷²⁹ 301–3°,⁸¹⁴ b_4 141°,¹³²⁶ b_{10} 159°,^{726a} b_{14} 164–5°,³⁶⁶ b_{15} 165–6°,⁷²⁵ $b_{16.5}$ 167°,²¹ 726b, ⁷²⁹ b_{17} 168–70°,⁵⁶⁹ b_{55} 199°, b_{126} 227°; ^{726a} d_0

- 1.3709,⁷²⁵ 1.3712,^{726b}, ⁷²⁹ $d_{15.2}$ 1.3554,⁷²⁵ 1.3561,^{726b}, ⁷²⁹ d 15/4
 1.338,^{974b} d 20/4 1.3515;¹³²⁶ n 15/D 1.6478,^{974b} n 20/D
 1.65002;¹³²⁶ surface tension 44.8 dynes/cm. at 16.5°, 37.84 at
 79.5°. ⁵⁶⁹
- p*-Nitrophenyl, $(O_2NC_6H_4)_2Se$, m . 175°, ¹⁰⁵² 171°. ⁶¹
- 2,4-Dinitrophenyl, $[(O_2N)_2C_6H_3]_2Se$, m . 196°, ^{88b} 194°. ¹³⁰⁹
- Benzyl, $(PhCH_2)_2Se$, m . 49°, ^{1094b} 45.5°, ⁶⁵³ 45°. ⁹⁶⁸
- Nitrobenzyl; $(O_2NC_6H_4CH_2)_2Se$, *ortho* m . 95°; *meta* m . 110°;
para m . 146°. ¹²⁰⁸
- o*-Tolyl, $(MeC_6H_4)_2Se$, m . 64°, ¹⁰¹⁷ 63°, ^{88a} 62°; ¹³⁹⁸ b_{13} 178–80°, ⁷⁹⁴
 b_{16} 186°. ¹³⁹⁸
- p*-Tolyl, $(MeC_6H_4)_2Se$, m . 70.5°, ²⁷⁶ 70°, ⁸⁹ 69.5°; b_{16} 196–6.5°. ¹³⁹⁸
- Naphthyl, $(C_{10}H_7)_2Se$, α m . 114°; ⁸¹⁵ β m . 138.5°; b_{15} 298°. ^{726a}
 $(\cdot CH_2SeCH_2Ph)_2$, m . 69°. ⁴⁶¹
- 1,3-bis(Ethylseleno)propane, $EtSeCH_2CH_2CH_2SeEt$, b_{15} 135°;
 d 20/4 1.4630; n_D 1.54892. ^{288b}
- β, β' -(Ethylselenoethyl) sulfide, $S(CH_2CH_2SeEt)_2$, m . 15°; b_4
 171°; d 25/4 1.4681. ¹¹⁷⁴

Unsymmetrical Selenides

- Methyl ethyl, $MeSeEt$, b . 86°; d 23/4 1.3134; n_D 1.4820. ^{288b}
- propyl, $MeSePr$, b . 114°; d 20.4/4 1.2445; n_D 1.4812. ^{288b}
- butyl, $MeSeBu$, b . 141°; d 24.5/4 1.1875; n_D 1.4771. ^{288b}
- decyl, $MeSeDec$, b_{14} 137–8°. ¹⁹⁸
- phenyl, $MeSePh$, b . 201°, ¹⁰¹⁵ b_{744} 202–3°; d 25/4 1.3954; n
 25/D 1.6039; surface tension 38.79 at 25°. ⁹³⁴
- nitrophenyl, $MeSeC_6H_4NO_2$, *ortho* m . 68°; ⁶⁷⁶ *meta* m . 31°;
 b_{13} 165°; ⁶¹ *para* m . 58.5°, ⁶⁷⁶ 56°. ⁶¹
- 6-nitro-*o*-tolyl, $MeSeC_6H_3(NO_2)Me$, b_1 121°. ⁶⁷⁶
- 2-nitro-*p*-tolyl, $MeSeC_6H_3(NO_2)Me$, m . 67.5°. ⁶⁷⁶
- naphthyl, $MeSeC_{10}H_7$, α b_{16} 173°; β m . 54°. ⁷⁹²
- Ethyl phenyl, $EtSePh$, b . 214°, ⁸⁹ 214–6°, ⁴³⁹ b_{748} 214–6°, ⁹³⁴ b_{20}
 120–4°; ⁸⁹ d 25/4 1.3167; n 25/D 1.5825; surface tension 37.25
 at 25°. ⁹³⁴
- *o*-nitrophenyl, $EtSeC_6H_4NO_2$, b_2 142–3°. ⁶⁷⁶
- α -naphthyl, $EtSeC_{10}H_7$, b_{13} 167–8°. ⁷⁹²
- Propyl phenyl, $PrSePh$, $b_{15.3}$ 117.7–8.2°; d 25/4 1.2641; n 25/D
 1.5691; surface tension 36.06 at 25°. ⁹³⁴
- i*-Propyl α -naphthyl, $Me_2CHSeC_{10}H_7$, b_{14} 165–7°. ⁷⁹²
- Butyl phenyl, $BuSePh$, b_5 105°, ⁴³⁸ $b_{15.3}$ 128.5–9.1°; d 25/4 1.2221;
 n 25/D 1.5589; surface tension 35.28 at 25°. ⁹³⁴

— naphthyl, $\text{BuSeC}_{10}\text{H}_7$, α b_{13} 180° ; β m. 137° .⁷⁹²
 Amyl phenyl, AmSePh , b_5 116° .⁴³⁸
i-Amylphenyl, $\text{Me}_2\text{CHCH}_2\text{CH}_2\text{SePh}$, b_3 105° .⁴³⁹
 Hexyl phenyl, HexSePh , b_5 133° ,⁴³⁸ $b_{18.3}$ 159.9° ; d 25/4 1.1609; n 25/D 1.5435; surface tension 34.04 at 25° .⁹³⁴
 Heptyl phenyl, HepSePh , $b_{15.7}$ 170.2° ; d 25/4 1.1367; n 25/D 1.5382; surface tension 33.63 at 25° .⁹³⁴
 Octyl phenyl, OctSePh , $b_{15.8}$ $182.3\text{--}3.3^\circ$; d 25/4 1.1173; n 25/D 1.5326; surface tension 33.25 at 25° .⁹³⁴
 Decyl phenyl, DecSePh , $b_{18.4}$ 212.4° ; d 25/4 1.0847; n 25/D 1.5250; surface tension 32.90 at 25° .⁹³⁴
 Phenyl *o*-nitrophenyl, $\text{PhSeC}_6\text{H}_4\text{NO}_2$, m. 92° .⁶⁷⁸
 — tolyl, $\text{PhSeC}_6\text{H}_4\text{Me}$, *ortho* m. 44° ; ⁶⁷⁷ *para* b_{20} $175\text{--}8^\circ$.⁴⁷⁸
 — *o*-ethylphenyl, $\text{PhSeC}_6\text{H}_4\text{Et}$, b_2 193° .⁶⁷⁷
 — α -naphthyl, $\text{PhSeC}_{10}\text{H}_7$, b_1 180° .²⁵¹
 Benzyl *p*-tolyl, $\text{PhCH}_2\text{SeC}_6\text{H}_4\text{Me}$, m. 33° .^{1261c}
 — α -naphthyl, $\text{PhCH}_2\text{SeC}_{10}\text{H}_7$, m. 69° .^{1261c}
p-Tolyl 2-biphenyl, $\text{MeC}_6\text{H}_4\text{SeC}_6\text{H}_4\text{Ph}$, m. 80° .²⁵¹

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CHAPTER 4.

Hydroxy- and Halo-Sulfides

Of all the substituted sulfides the hydroxysulfides and chlorosulfides exhibit the most interesting properties and have been most extensively exploited. Logically these do not belong together, but practically they do since, in most cases, the hydroxysulfides have been prepared as intermediates on the way to the chlorosulfides.

Attention has been focused on the chlorosulfides by the introduction of β,β' -dichloroethyl sulfide as a war gas. In both classes the interest is centered on those in which the substituent is in the beta position to the sulfur atom. The hydroxy- and chlorosulfides, $\text{RSCH}_2\text{CH}_2\text{OH}$ and $\text{RSCH}_2\text{CH}_2\text{Cl}$, have peculiar reactivity due to the labilizing influence of the sulfur atom in the beta position.

Hydroxyalkyl Sulfides, $\text{RS}(\text{CH}_2)_n\text{OH}$

α -Hydroxyalkyl Sulfides, RSCH_2OH

The lowest members of this class are RSCH_2OH in which $n = 1$. When a mercaptan and formaldehyde are brought together, an addition compound is formed:

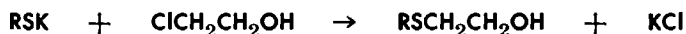


These products are too unstable to be definitely characterized. They are regarded as intermediates in the formation of mercaptals.^{848b} * See the chapter on mercaptals. The acetate has been made from chloromethyl acetate, $\text{MeCO}_2\text{CH}_2\text{Cl}$, and methyl mercaptan.^{780a} The hydroxymethyl benzyl sulfide, $\text{PhCH}_2\text{SCH}_2\text{OH}$,^{150.5} and the acetate of PhSCH_2OH ¹¹³⁵ are known. Chloral is peculiar in that the hemiacetals $\text{Cl}_3\text{CCH}(\text{OH})\text{SR}$, like the alcoholates, $\text{Cl}_3\text{CCH}(\text{OH})\text{OR}$, are stable.^{733.5, 923.5, 956d, 995.5, 1115}

β-Hydroxyalkyl Sulfides, $\text{RSCH}_2\text{CH}_2\text{OH}$

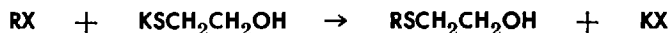
This is the most interesting group and has the largest number of members. It is also the one for which the starting materials are the most readily available.

The obvious method is the reaction of ethylene chlorhydrin with a mercaptan in alkaline solution.^{367, 504, 825, 1339}



A weighed amount of sodium, or sodium methylate,¹¹⁵⁸ is dissolved in absolute alcohol and the calculated amount of mercaptan poured into the cooled solution. The formation of the sodium mercaptide is instantaneous and complete. Without delay the chlorhydrin is added with shaking. The reaction is rapid, as is shown by the precipitation of sodium chloride, but it is well to heat for a few minutes. The sodium chloride is filtered off, the alcohol stripped out, and the residue fractionated. Sodium, or potassium, hydroxide in absolute or even 95% alcohol, may be used, but the separation of the salt is not quite so complete.¹¹⁵⁸ The β -hydroxypropyl compounds, $\text{MeSCH}_2\text{CH}(\text{OH})\text{Me}$ ⁶⁸⁴ and $\text{EtSCH}_2\text{CH}(\text{OH})\text{Me}$,^{1430c} are from propylene halohydrins.

The reverse reaction, an alkyl halide with mercaptoethanol, gives the same product:



Since mercaptoethanol has become available, this is more convenient.

The methyl compound, $\text{MeSCH}_2\text{CH}_2\text{OH}$, has received much attention since it is an intermediate in one synthesis of methio-

* As a large proportion of the authors quoted in Chapters 4, 5, 6 and 7 have contributed to two or more of these chapters, the literature cited for all four is combined and placed at the end of Chapter 7.

nine.^{164, 780a, 901, 1468, 1469} Potassium hydroxide is dissolved in ethanol, the equivalent amount of mercaptoethanol is added, and methyl chloride, or bromide, vapor is passed in. The salt is filtered off and the filtrate fractionated.¹¹⁶⁰ Somewhat neater results are obtained if water is excluded entirely as indicated previously.

Many compounds of the general formula $\text{RSCH}_2\text{CH}_2\text{OH}$ have been reported, in which R is ethyl,^{127, 367, 956c, 1277, 1281, 1323} butyl,^{127, 1455a} *i*-butyl,²²⁰ *s*-butyl,⁶⁸⁹ *t*-butyl,^{350d, 689} *i*-amyl,¹²⁷⁷ allyl,¹²⁷⁶ heptyl, nonyl, undecyl, methoxyethyl, propoxyethyl, butoxyethyl,⁵⁷⁵ phenyl,^{480, 781, 1277} a substituted phenyl,^{66a, 106a, 506} or γ -diethylaminopropyl.^{556b} A terpinyl derivative has been claimed as a textile assistant.^{581, 1302c}

Two selenium compounds, $\text{PhSeCH}_2\text{CH}_2\text{OH}$ and $\text{PhCH}_2\text{SeCH}_2\text{CH}_2\text{OH}$, have been reported.⁸⁹²

Pentene chlorhydrin, $\text{PrCH}(\text{OH})\text{CH}_2\text{Cl}$, and methyl mercaptan give the hydroxysulfide, $\text{MeSCH}_2\text{CH}(\text{OH})\text{Pr}$.⁵⁶⁹

Another good way to obtain a β -hydroxyethyl sulfide is to cause ethylene oxide to react with a mercaptan: ^{309, 330, 331b, 383, 414a, 504, 698, 1034b, 1271, 1316c, 1325}



An alkaline catalyst is required and care must be used to keep the reaction from becoming violent.⁹⁴⁷ If an excess of the ethylene oxide is used, waxy materials, $\text{RS}(\text{CH}_2\text{CH}_2\text{O})_n\text{CH}_2\text{CH}_2\text{OH}$, are obtained.^{339, 390a, 693a, 993, 1058b, 1235} Similar compounds of the general formula $\text{RSCH}_2\text{O}(\text{CH}_2\text{CH}_2\text{O})_n\text{CH}_2\text{CH}_2\text{OH}$ are made from a monochloromethyl ether of a polyoxyethyleneglycol and a mercaptide.⁵⁴¹

An unsymmetrical epoxide gives a β -hydroxyalkylsulfide.^{166a, 513} Thus propylene oxide gives a β -hydroxypropylsulfide, $\text{RSCH}_2\text{CH}(\text{OH})\text{Me}$.^{627a, 630, 924.5, 1257, 1258} With epichlorhydrin, the product is $\text{RSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{Cl}$.^{1034b, 1391} This may be converted to an epoxide which can react with another mercaptan,¹³⁹¹ an alcohol,¹¹⁸¹ or an amine.¹³⁹² The reaction of an alkylmercaptopropylene oxide with an alcohol gives polymers, $\text{RO}[\text{CH}_2\text{CH}(\text{CH}_2\text{SR}')\text{O}]_n\text{H}$ which may go into synthetic lubricants.⁹⁷¹

Styrene oxide and butadiene monoxide react with mercaptans, in the presence of a base, to give compounds of the types, $\text{PhCH}(\text{OH})\text{CH}_2\text{SR}$ and $\text{CH}_2:\text{CHCH}(\text{OH})\text{CH}_2\text{SR}$.⁵⁵⁸ The same PhCH -

(OH)CH₂SPr has been obtained by shaking styrene and propyl mercaptan with oxygen.⁷⁶⁴

Trimethylene oxide reacts less rapidly but in the same way as ethylene oxide:¹²⁶⁰



The simplest possible way to prepare a β -hydroxyethyl sulfide is the addition of mercaptoethanol to an unsaturate:^{335, 1047a, 1158}



If thioglycerol is the addendum, the product is a sulfideglycol.^{581, 1303} A γ -hydroxy sulfide is obtained when allyl alcohol is the unsaturate:^{674.5, 746, 1347}



The traces of peroxides usually present are sufficient to catalyze this reaction. Triton-B, potassium carbonate, and tetra(hydroxyethyl)ammonium hydroxide are recommended as catalysts.¹³⁴⁷ Mercury methyl mercaptide is a catalyst for the addition of methanethiol to allyl alcohol, but it acts best in the presence of oxygen and of light.⁷⁴⁶ These additions go contrary to Markownikow's rule, but in the presence of sulfur the addition of thiophenol to allyl alcohol is according to this rule.⁵¹⁹

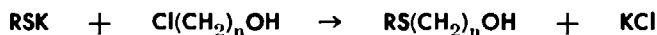
An alternative method is to add a mercaptan to vinyl acetate and hydrolyze the product:^{442.5, 674.5, 1347}



Mercaptans may be added to dihydropyran. The product is a 2-alkylmercaptopentamethylene oxide.^{778d}

Other Hydroxyalkyl Sulfides, RS(CH₂)_nOH

A number of these, with values for n from 2 to 18, have been prepared by the reaction of a halohydrin or its acetate on a mercaptide:^{106b, 107, 109, 112a, 112b, 112c, 113, 244, 780a, 1193, 1211a}



γ -Methylmercaptopropanol has been isolated from soy sauce.^{10a, 10b} It has been synthesized from trimethylene chlorhydrin and sodium methyl mercaptide and also by methylating γ -mercapto-propanol.^{11, 111c}

Esters of polythioglycols have been compared with other proposed synthetic lubricants.⁸²⁶

The hydroxy-*bis*-sulfide, $(\text{PhCH}_2\text{SCH}_2)_2\text{CHOH}$, has been made from epichlorhydrin and sodium benzyl mercaptide.⁵⁰⁵ Sodium *p*-thiocresolate may react with the halogen without opening the epoxide ring.^{331a}

Ether-Sulfides

These are formed readily by the addition of a mercaptan to a vinyl ether: ^{442.5, 1284}



As was brought out in Chapter 2, Volume I, the addition of mercaptans goes contrary to Markownikow's rule in the presence of oxygen, peroxides, or of bases. The amounts of peroxides commonly present are usually sufficient.¹¹⁵⁸ In the presence of sulfur dioxide, the addition goes the other way: ¹²⁸⁴



The product is a monothioacetal.

Reactions

The sulfide alcohols, $\text{RS}(\text{CH}_2)_n\text{OH}$, have two sets of reactions. As sulfides, they can be oxidised to sulfoxides and sulfones.^{466a, 627d, 825, 1339} They can combine with alkyl halides to form sulfonium salts^{196, 1118} and can form complexes with salts of mercury and of other heavy metals. These reactions have not been exploited extensively, but it is known that such reactions take place and that they are modified somewhat by the presence of the hydroxyl group. When the hydroxyl is in the β -position, as in $\text{RSCH}_2\text{CH}_2\text{OH}$, oxidation by nitric acid goes all the way to the sulfonic acid, RSO_3H .^{466b} Oxidation by chlorine gives the sulfone, with replacement of the hydroxyl by chlorine.^{441.7} $\text{EtSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{SEt}$ is oxidised to the *bis*-sulfone by hydrogen peroxide.^{1211b}

These sulfide alcohols give also the characteristic reactions of alcohols though these are more or less modified by the presence of the sulfur atom. Its influence is particularly great when it is in the β -position to the hydroxyl. The alcohols, $\text{RSCH}_2\text{CH}_2\text{OH}$, resemble tertiary alcohols in that the bond between the hydroxyl

and carbon is weak. To replace the hydroxyl by a halogen, it is not always necessary to use such reagents as thionyl chloride; simply heating with concentrated aqueous hydrochloric acid may be sufficient. Thus β -butylmercaptoethanol, $\text{BuSCH}_2\text{CH}_2\text{OH}$, is converted to the chloride, $\text{BuSCH}_2\text{CH}_2\text{Cl}$, by refluxing it 4 hours with concentrated hydrochloric acid.^{1455a}

In other positions, the sulfur atom influences the reactivity of the hydroxyl group but to a far less degree. Several of the phenyl sulfide alcohols, $\text{PhS}(\text{CH}_2)_n\text{OH}$, were heated, in phenol as a solvent, with equivalent amounts of hydrogen bromide. The rate of conversion to the bromide was equal to that of cetyl alcohol when n was 9 or 10, somewhat greater when n was 6, 7 or 8, less when it was 3, and far greater when it was 2 or 4. The hydrolysis velocities of the halides, $\text{RSCH}_2\text{CH}_2\text{X}$, bear a similar relation to one another.^{112a, 112b}

Information about the esterification of sulfide alcohols is scanty. It is probable that the ease of esterification and the stability of the esters will be found to vary with the distance of the hydroxyl from the sulfur atom. Certain esters have been patented.^{1275, 1281} β -Hydroxyethyl sulfide, $\text{EtSCH}_2\text{CH}_2\text{OH}$, and *p*-nitrobenzoyl chloride, in pyridine, give the *p*-nitrobenzoic ester which has been reduced to the amino ester.²⁴⁸ The benzoate of $\text{MeSCH}_2\text{CH}_2\text{OH}$ has been made similarly.³²⁵ Many esters have been prepared by using an ester as one of the original reactants.⁸⁰⁰

Treating methyl phenyl sulfide and benzal chloride with aluminum chloride gives *p*-methylmercaptotriphenylcarbinol, $\text{MeSC}_6\text{H}_4\text{CPh}_2\text{OH}$. From benzotrichloride the product is $(\text{MeSC}_6\text{H}_4)_2\text{CPhOH}$.¹⁹⁴

A mercaptan, containing hydroxyl groups, may be condensed with an *N*-methylolamide.^{1302d}

Uses

The toxicities and repellent powers of several β -hydroxyethyl alkyl sulfides have been studied.^{414b, 867, 1316c} Some of them have been claimed as disinfectants^{629, 630} and as useful in soaps, creams, and emulsions.^{627c} One from 2-mercaptobenzothiazole and ethylene oxide is said to be a pickling inhibitor.⁹⁹³ Another from the addition of mercaptoethanol to styrene is claimed as a fungicide.³⁵⁵ Those having long alkyl groups are said to be frothing and dispersing agents.^{309, 1271} By starting with long-

chain mercaptans and using ethylene chlorhydrin or epichlorhydrin, products are obtained which may be useful as such or which may be sulfated or otherwise treated.^{72, 373, 627b, 628, 692c, 705b, 706, 783, 835, 1302a}

Hydroxypolyether sulfides, $RS(CH_2CH_2O)_nCH_2CH_2OH$, are claimed as surface-active agents,^{339, 541} as detergents,^{1058b} and as additions to viscose.¹²³⁵

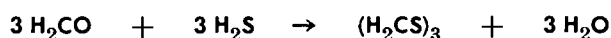
The reaction of mercaptides with polyhalogen compounds, containing also one or more hydroxyls, is said to give useful compounds.⁸⁵⁹

An oil-soluble hydroxylated sulfide is said to be obtained by adding a sulfur chloride to cracked paraffin wax and hydrolyzing.¹⁴³⁷

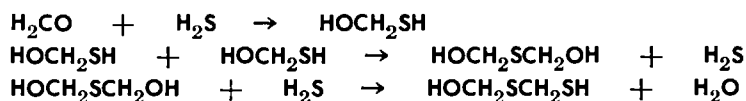
Sulfide Glycols, $HO(CH_2)_nS(CH_2)_mOH$

The lowest member of this class is dihydroxymethyl sulfide, $HOCH_2SCH_2OH$, in which $n = m = 1$. This has never been isolated, but is believed to be formed transiently in the hydrolysis of the chloride, $ClCH_2SCH_2Cl$. Its dimethyl ether, $MeOCH_2SCH_2OMe$, is obtained when this chloride is added to methanol containing alkali.¹⁴²

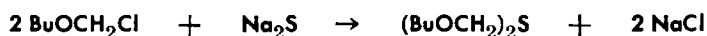
The formation of trithiane from formaldehyde and hydrogen sulfide, in the presence of acid, may be written:



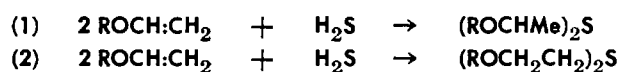
The first steps in this synthesis may be:



An alkoxymethyl sulfide is obtained when a chloromethyl ether reacts with sodium sulfide:^{1282, 1283}



The addition of hydrogen sulfide to a vinyl ether may go in either of two ways:

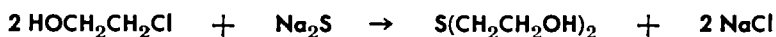


A mixture of the two products may be obtained.^{1126, 1284} The conditions determine the mode of addition. As with mercaptans,

the addition goes according to Markownikow's rule in the presence of sulfur dioxide, reaction (1), but with a trace of oxygen, the abnormal product is formed according to reaction (2).¹²⁸⁴

Thiodiglycol, S(CH₂CH₂OH)₂

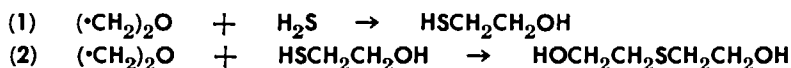
This compound has been of special interest on account of its relation to mustard gas. It was first obtained by Carius²⁵⁴ as a by-product in making monothioglycol, HSCH₂CH₂OH. Victor Meyer^{956b} prepared it from ethylene chlorhydrin and sodium sulfide:



During the first World War, the Germans manufactured many tons of it by this method. It was found by Gomberg⁵⁷⁷ that much less sodium sulfide is required than was called for in the original directions and that the easily available 20% ethylene chlorhydrin serves as well as the 100%. The laboratory preparation has been fully described.^{436, 577} Calcium sulfide has been recommended instead of the sodium.¹⁶

The making of thiodiglycol by this method is easy and the yield is high, but getting it away from the sodium chloride that is formed and the sodium sulfide that may be left over is not a simple matter.

Recently it has been found that it can be made easily and in 90% yield from ethylene oxide and hydrogen sulfide. These unite in the presence of alkali as a catalyst.^{283, 284, 390b, 1034b, 1034c, 1064, 1231, 1482b, 1482c} There are two steps:



The two steps take place under the same conditions and the one follows the other quickly. The second reaction appears to be more rapid than the first, but this may be due to the relatively low solubility of the hydrogen sulfide. Actually the product always contains monothioglycol and thiodiglycol, the proportions of which vary according to the relative rates at which the two reactants are introduced. Thiodiglycol, containing some sodium ethylate, is heated at 50 to 60° and hydrogen sulfide and ethylene oxide are passed in separately beneath the surface. A study has been made of the kinetics of this reaction and of the

similar one with propylene oxide.¹¹⁹ The mercaptoethanol can be converted to thiodiglycol by heating it with ethylene carbonate or sulfite.²⁵⁵ Thiodiglycol is formed along with some of the disulfide when ethylene oxide and carbon dioxide are passed into sodium polysulfide solution.^{441.3}

Thiodiglycol is a somewhat viscous liquid resembling diethylene glycol in appearance. It mixes with water, the lower alcohols, chloroform, and ethyl acetate in all proportions. At 25° its solubility in 100 g. of solvent is 1.07 g. for benzene, 7.09 g. for anhydrous ether and 0.06 g. for ligroin. Alone, or with an organic acid, it is stable at 180° for many hours. With dilute alkali, the sulfide ion is formed slowly at 100°.²⁹⁵ A curious property is its power to dissolve silver salts; 100 g. dissolves 17.16 g. silver chloride. For this reason, low results are obtained when the chloride ion is titrated in its presence.¹⁰⁰⁴ It is said to be a catalyst for the decomposition of formamide into carbon monoxide and ammonia.⁴²⁵ Heated with potassium bisulfate, it is dehydrated to thioxane.⁵⁰⁸ It is readily oxidised by bromine. In sulfuric acid, it reacts with iodates and bromates like a sulfite.^{668a}

The ultraviolet absorption of its vapor,⁷⁰⁴ its infrared spectrum, and that of its acetate in solvents have been studied.¹²⁵³

Hydrolysis of the bromine addition product, $\text{Br}_2\text{S}(\text{CH}_2\text{CH}_2\text{OH})_2$, gives the sulfoxide.⁵⁰⁶ This is formed in 20 days when a solution of thiodiglycol in citral is exposed to the air.¹⁷⁴

By saponification of the petroleum-ether extract of the adrenal cortex, thiodiglycol sulfoxide has been isolated. It is supposed to be present as an ester of a higher fatty acid.^{1156, 1157}

Oxidation of thiodiglycol with perbenzoic acid leads to the sulfoxide, $\text{OS}(\text{CH}_2\text{CH}_2\text{OH})_2$, m. 112°, or to the sulfone, $\text{O}_2\text{S}(\text{CH}_2\text{CH}_2\text{OH})_2$, m. 58°, according to conditions.^{850b} The oxidation may be effected by iodosobenzene, by 30% hydrogen peroxide in acetic acid,^{479a, 479b} or by ozone in water solution.¹¹²⁰ The rate of oxidation has been measured.¹²⁰⁵ The reaction is followed by titration with standard hypochlorite solution.⁸³⁹ Several other oxidising agents may be used similarly. Amperometric titration with electrolytic bromine is accurate within 2%.¹²⁶² With potassium iodoplatinate, the determination can be made colorimetrically.¹²⁶¹

Thiodiglycol is esterified by heating with organic acids or

their anhydrides.^{7, 295, 443c, 802a, 1037, 1106} The esterification with heptoic, lauric, and adipic acid has been studied with continuous removal of the water. The reaction is initially of the second order. In the presence of a strong acid as catalyst, it continues of this order to the end. At 177° the esterification of $S(CH_2CH_2OH)_2$ is like that of $O(CH_2CH_2OH)_2$.³⁰⁷ The acetate has been made from mustard gas and sodium acetate.⁶²⁶ The esters from caproates to caprates have been prepared by heating thiodiglycol with slightly more than two equivalents of the acid at 160 to 180° for 15 hours under a slight vacuum with no catalyst. With dibasic acids, such as succinic, adipic, azelaic, and sebacic, an excess of the thiodiglycol was used. This excess was removed by bubbling nitrogen through the polyester at 200 to 250°. Molecular weights of several thousand have been attained.³⁰⁷

Acid catalysts cause self-condensation of thiodiglycol:



Continued heating with an acid catalyst converts thiodiglycol into polymers which are recommended for several uses.^{76a}

As water is formed in the etherification, as well as in the esterification, taking off 2 moles of water from a mixture of 1 mole of thiodiglycol and 2 moles of an acid, in the presence of an acid catalyst, does not mean the formation of 100% of the neutral ester.¹¹⁵⁸

As is shown in Table 1.4, the acid hydrolysis of the acetate of thiodiglycol is slower than that of ethyl acetate, but the alkaline is much faster.³⁴⁷

TABLE 1.4
Relative Rates of Hydrolysis of the Acetates

	Acid	Alkaline
EtOH	3.28	1900
$S(CH_2CH_2OH)_2$	2.36	8100
$EtSCH_2CH_2OH$	2.38	5800

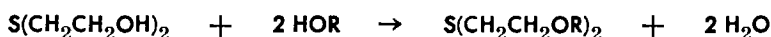
The monoacetate has been prepared by adding mercapto-ethanol to vinyl acetate:^{1216, 1347}



The benzoate can be made by shaking a cold, alkaline solution of thiodiglycol with benzoyl chloride. It can be oxidised to the sulfoxide and the sulfone.⁵⁰⁶ The *p*-nitrobenzoate and its sulfone have been reduced to the corresponding amino compounds.⁹⁰⁵

Thiodiglycol esters, particularly those from dibasic acids, have been claimed to be useful in plastics.¹⁴⁴³ Esters are said to be plasticizers for synthetic resins^{68.5, 1106} and additions to high-pressure lubricants.⁴⁴⁴ The esters of dibasic acids are said to be rubberlike.^{693c, 1443} The sulfurized esters of unsaturated acids, such as oleic, are claimed as additions to high-pressure lubricants.^{139c}

Ethers of thiodiglycol are formed by heating it with primary alcohols:

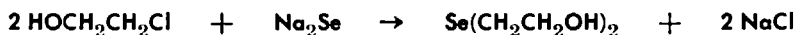


An acid catalyst is used. As the hydroxyl of the thiodiglycol and the hydrogen of the primary alcohol are labile, the etherification goes in one direction only.^{1175, 1176} With pentaerythritol as the primary alcohol, the products are plastics.⁴⁴ Two of these ethers, $\text{S}(\text{CH}_2\text{CH}_2\text{OEt})_2$ and $\text{S}(\text{CH}_2\text{CH}_2\text{OBu})_2$, were among the products obtained in the addition of hydrogen sulfide to the vinyl ethers, EtOCH:CH_2 and BuOCH:CH_2 .¹¹²⁶ This is a general reaction with vinyl alkyl ethers.^{693b, 1126, 1284} The diethyl ether, $\text{S}(\text{CH}_2\text{CH}_2\text{OEt})_2$, has been found among the products of the hydrolysis of mustard gas by alcoholic potash.³⁴⁰ It is one of several products formed in the reaction of zinc with mustard gas in alcoholic solution. The methyl, propyl, butyl, and *i*-amyl ethers are produced similarly when the corresponding alcohols are solvents.^{808a}

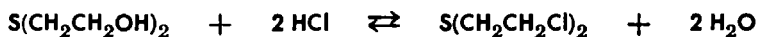
The monoethyl ether, $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OEt}$, is one of the products when mustard gas is hydrolyzed by alkali in dilute alcohol.³⁴⁰ It is formed by the addition of mercaptoethanol to vinyl ethyl ether, EtOCH:CH_2 . The *i*-propyl and *n*-butyl monoethers, $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OCHMe}_2$ and $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OBu}$, are prepared similarly.³⁵²

When thiodiglycol vapor, with or without hydrogen sulfide, is passed over alumina at 200 to 275°, dithiane and thioxane are formed.¹⁴⁹³ Dithiane is produced when thiodiglycol is heated to 180° with hydrobromic acid.¹⁰¹

The selenium analog has been prepared: ¹¹⁰⁴



The conversion of thiodiglycol to mustard gas and the reverse hydrolysis are very simply represented by the equation:



This does show the end products, but says nothing about the intermediate stages, which are known to be sulfonium complexes.^{340, 430} They break down as the reaction progresses. These will be considered more fully later as reactions of mustard gas.

The following analogous sulfonium compounds have been prepared by heating together the required components:

$\text{Et}_2(\text{HOCH}_2\text{CH}_2)\text{S}\cdot\text{Cl}$, bromide, iodide m. 97°

$\text{Et}(\text{HOCH}_2\text{CH}_2)_2\text{S}\cdot\text{Cl}$, m. 119°, bromide m. 85°, iodide decomposed at 65°

$(\text{HOCH}_2\text{CH}_2)_3\text{S}\cdot\text{Cl}$, m. 127°, bromide m. 104°, iodide decomposed at 65°

Solutions of the free bases can be prepared by means of silver oxide, but they decompose on concentration. The hydroxyls in these compounds can be replaced by chlorine with thionyl chloride but not with hydrogen chloride. The trichlorotriethyl sulfonium chloride, $(\text{ClCH}_2\text{CH}_2)_3\text{S}\cdot\text{Cl}$, is a hygroscopic solid and has no vesicant action on the skin.²⁹⁵

Thiodiglycol unites with such halides as ethyl bromide and benzyl chloride to form water-soluble sulfonium complexes which are said to be useful in dyeing.^{692d} Various substituted benzyl chlorides may be used.²¹⁵ Thiodiglycol and dimethyl sulfate form the methyl sulfate, $\text{Me}(\text{HOCH}_2\text{CH}_2)_2\text{S}\cdot\text{OSO}_3\text{Me}$, m. 50°.²⁷⁹ The sulfonium salt with *p*-fluorophenacyl chloride melts at 115°.^{69.5}

Thiodiglycol is manufactured on a moderate scale and sold under the name of "Kromfax solvent." It is recommended as a solvent in the application of certain dyes,^{443b} in printing,⁸⁸⁷ and also as a selective solvent in the extraction of butadiene.¹⁴⁴⁴

Thiodiglycol prevents the clotting of blood.⁴⁶⁴ Its toxicity to rabbits's cornea⁶⁸¹ and to insects has been studied.^{250, 867} Its physiological effects have been compared to those of mustard gas.^{32, 790, 1089d}

Other Sulfide Glycols

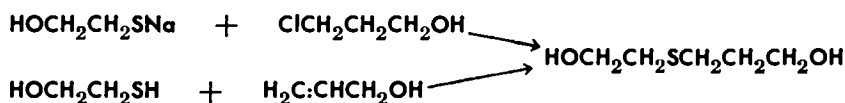
Glycols with One Sulfide Link

There are three β -hydroxyethyl hydroxypropyl sulfides:

- (1) $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{OH}$
- (2) $\text{HOCH}_2\text{CH}_2\text{SCHMeCH}_2\text{OH}$
- (3) $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH(OH)CH}_3$

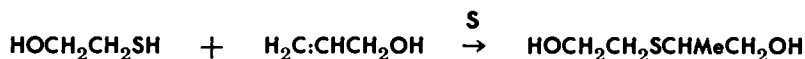
In (2) and (3) both of the hydroxyl groups are on the β -carbon atom. If the hydroxyl were on an α -carbon the compound would be an unstable hemimercaptal. Glycols (2) and (3) have been involved in the study of the mechanism of mustard-gas formation from ethylene and sulfur chloride.⁵²³ In spite of the work that has been done on them, several things about them remain obscure.

Glycol (1) has been prepared in the standard way from mercaptoethanol and 3-chloropropanol^{352, 599} and by the addition of mercaptoethanol to allyl alcohol: ^{352, 523, 581, 599}

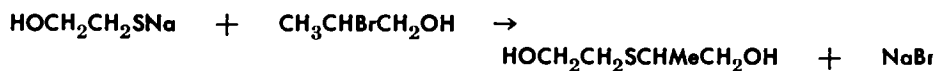


The second reaction, like other addition reactions, is influenced greatly by various factors, some not understood and some unknown. It is described as taking place only on refluxing and then with a 50% yield⁵⁹⁹ and as going spontaneously,^{352, 581} the temperature rising 50° within a minute after mixing with a practically quantitative yield.³⁵²

Glycol (2) has been prepared by the addition of mercaptoethanol to allyl alcohol in the presence of sulfur,^{523, 599} which is known to favor addition according to Markownikow's rule:⁷³⁷

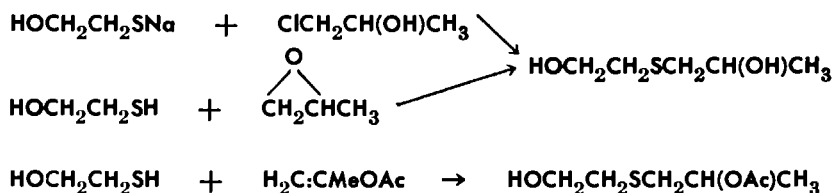


Two attempts have been made to prepare it synthetically.^{352, 1462} The reaction of 2-bromopropanol with mercaptoethanol in alkaline solution was expected to give it:



The product turned out to be glycol (3) as shown by its density and refractive index.³⁵² The reaction of 2-chloropropanol with potassium phthalimide gives $\text{C}_6\text{H}_4(\text{CO})_2\text{NCH}_2\text{CH}(\text{OH})\text{CH}_3$ ⁵²⁹ instead of the expected isopropyl derivative; this was explained by assuming propylene oxide as an intermediate.

Glycol (3) has been prepared in three ways, all of which give the same product. Mercaptoethanol reacts with propylene chlorhydrin^{352, 599} or with the oxide.¹⁰⁶⁸ It can be added to isopropenyl acetate and the product hydrolyzed:



Alcoholysis of the acetate gives the glycol.³⁵²

The two sulfide-glycols, $\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_4\text{OH}$ and $\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_5\text{OH}$, have been made starting with mercaptoethanol and the acetates of δ -chlorobutyl and ϵ -chloroamyl alcohols.³⁵²

γ, γ' -Dihydroxypropyl sulfide, $\text{S}(\text{CH}_2\text{CH}_2\text{CH}_2\text{OH})_2$, has been prepared from a trimethylene halohydrin and sodium sulfide. Two different melting points, 148° ^{111a} and 123° ,⁸⁹⁰ have been given for its phenylurethane. The same glycol has been made from trimethylene oxide and sodium sulfide. When prepared in this manner, its phenylurethane melts at 120° .¹²⁶⁰ This glycol is formed when hydrogen sulfide is added to allyl alcohol under the influence of ultraviolet light^{608, 1410b, 1454} and when the ether, $(\text{PhCH}_2\text{OCH}_2\text{CH}_2\text{CH}_2)_2\text{S}$, is split by hydrogen bromide.^{111b} Its butyrate is formed when hydrogen sulfide is added to allyl butyrate.^{281.5} The hydrogen sulfide may come from ammonium or an alkylammonium hydrosulfide.^{68b} In its conversion to the chloride, it acts like an ordinary alcohol and not like thiodiglycol. The acetate, propionate, and other esters are said to be useful in lubricants.^{76b}

β, γ' -Dihydroxypropyl sulfide, $\text{HOCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}(\text{OH})\text{CH}_3$, has been obtained from γ -hydroxypropyl mercaptan and propylene chlorhydrin.¹¹⁹²

β, β' -Dihydroxypropyl sulfide, $\text{MeCH}(\text{OH})\text{CH}_2\text{SCH}_2\text{CH}(\text{OH})\text{Me}$, has been made from propylene chlorhydrin and sodium sulfide^{303, 802b, 924.5, 1192} and also from propylene oxide and hydrogen

sulfide.^{119, 924.5, 1462} It has been obtained by hydrolyzing the dichloride which is formed by adding sulfur chloride to propylene.³⁰³ It is said to have high solvent power for dyes.^{802b}

β,β' -Dihydroxy- β,β' -dimethylbutyl sulfide, $\text{EtCMe}(\text{OH})\text{CH}_2\text{SCH}_2\text{CMe}(\text{OH})\text{Et}$, has been prepared from the corresponding chloride and sodium sulfide.⁹⁰⁵ Tertiary sulfide alcohols, $[\text{PhCR}(\text{OH})\text{CH}_2]_2\text{S}$, can be made from a Grignard reagent and a ketosulfide, $(\text{PhCOCH}_2)_2\text{S}$.¹¹⁰¹

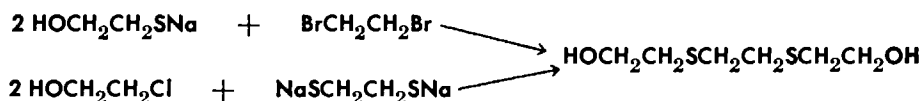
1-Hydroxy-2-cyclopentyl, 1-hydroxy-2-cyclohexyl, and 1-hydroxy-2-cycloheptyl sulfides have been prepared from the corresponding chlorides and sodium sulfide, also some more complicated ones of this class.^{1005a, 1006}

Two molecules of dibutyl phosphite may be added to one of allyl sulfide.^{1047b}

Polymers may be obtained by condensing an alkylene glycol with a dihydroxyalkyl sulfide¹⁰⁰² or from the reaction of epichlorhydrin with a dithiol.²⁵⁸ There are several examples of the use of hydroxysulfides as starting materials for making wetting agents.^{218, 835}

Glycols with Two or More Sulfide Links

The glycol $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$ has been made in two ways: ^{114, 530, 1200}



Its diethyl ether is one of the products when mustard gas is treated with zinc in ethanol.^{808a} The glycol is oxidised by hydrogen peroxide to the *bis*-sulfoxide, of which there are two forms— α , m. 139° and β , m. 105° —and finally to the *bis*-sulfone, m. 134.5° .^{1123a} The series, $\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_n\text{SCH}_2\text{CH}_2\text{OH}$, has been extended up to the decamethylene.⁵³⁰

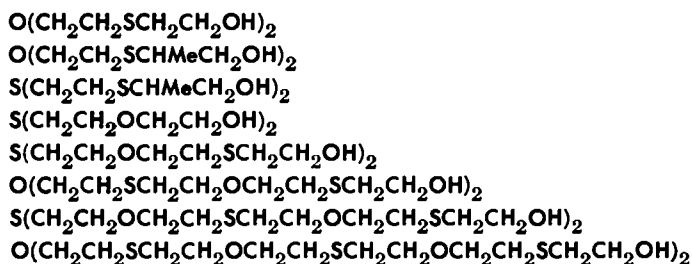
A number of sulfide glycols of this class have been made.

- (1) $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$ ^{101, 114, 1152, 1462}
- (2) $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$ ^{1152, 1319}
- (3) $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$ ¹¹⁵²
- (4) $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{S}(\text{CH}_2)_4\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$ ¹¹⁵²
- (5) $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{S}(\text{CH}_2)_5\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$ ¹¹⁵²
- (6) $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SO}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$ ^{1152, 1311, 1462}
- (7) $\text{HOCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{OH}$ ^{352, 722}

- (8) $\text{HOCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{OH}$ ¹¹⁵²
 (9) $\text{CH}_3\text{CH}(\text{OH})\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}(\text{OH})\text{CH}_3$ ^{352, 530, 599}
 (10) $\text{HOCH}_2\text{CHMeSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCHMeCH}_2\text{OH}$ ^{352, 1462}
 (11) $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SO}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$ ¹¹⁵²

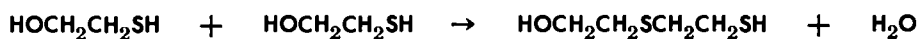
Glycol (1) was made in two ways: from mustard gas and mercaptoethanol ¹¹⁴ and from ethylene chlorhydrin and dimercaptoethyl sulfide. ¹¹⁵² Glycols (1), (2), (3), (5), (6), and (11) were made from ethylene chlorhydrin and the dimercaptan. ¹¹⁵² Glycols (9) and (10) were made similarly from propylene chlorhydrin ³⁵² and (8) from 3-chloropropanol. ¹¹⁵² Glycol (7) was from the addition of ethanedithiol to allyl alcohol. ^{352, 722} Glycol (6) was made from the reaction of mercaptoethanol with mustard sulfone ^{1152, 1311} or by its addition to vinyl sulfone. ¹³¹¹

Hydroxysulfides having ether as well as sulfide linkages are known. Two of these, $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$ and $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$, are readily obtained by the spontaneous addition of mercaptoethanol to divinyl ether and to diallyl ether, respectively. ³⁵² These and more complicated ones have been made by repeating conventional reactions with dichloroethyl ether as a starting material: ^{212, 1462, 1482a}



Polymeric adducts of hydrogen sulfide to allyl ether, boiling above 235° at 2 mm., are said to prevent the cracking of rubber. ⁸⁹⁶ Terminally unsaturated polysulfide polyethers are claimed as plasticizers for vinyl polymers. ^{1410c}

Mercaptoethanol, being an alcohol in which the hydroxyl is readily replaced and also a mercaptan, can condense with itself:



This may go on indefinitely with the formation of a polymer, $\text{HO}(\text{CH}_2\text{CH}_2\text{S})_n\text{CH}_2\text{CH}_2\text{SH}$. In one experiment, in which mercaptoethanol was heated at 100° with a catalytic amount of

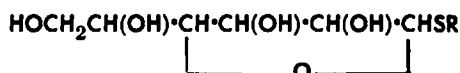
p-toluene sulfonic acid, a solid was produced whose sulfur content indicated an average value of 15 for n .¹¹⁵⁸ High polymers are formed if the heating is prolonged.

From dichloroethyl ether and a phenyl mercaptide, two products have been obtained, $\text{PhSCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{Cl}$ and $(\text{PhSCH}_2\text{CH}_2)_2\text{O}$.³²⁸ The ether-sulfide, $(\text{AmSCH}_2\text{CH}_2)_2\text{O}$, has been recommended as a lubricant for precision instruments.⁸² β -Decylmercaptoethanol, from decyl mercaptan and ethylene oxide, is converted to the ether-sulfide, $(\text{C}_{10}\text{H}_{21}\text{SCH}_2\text{CH}_2)_2\text{O}$, by sulfuric acid.³⁸³

SULFIDES OF THE SUGAR GROUP

This subject is complex and can be treated here only in outline. The subject has been reviewed.^{1147b} There are a few symmetrical and many unsymmetrical sulfides. Glucosyl, galactosyl and cellosyl sulfides and selenides have been prepared from the bromides and potassium sulfide or selenide.^{1017, 1240, 1243, 1483b, 1485}

Of the unsymmetrical sulfides, the most important are the thioglucosides:

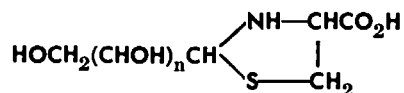


These are so named in accordance with sugar terminology. As sulfides, they can be made from glucothiose and an alkyl halide or from bromoglucose and a metal mercaptide. β -Ethylthioglucoside has been prepared in both ways.¹²⁴¹ Thioglucosides are closely related to the mercaptals of a later chapter. For ease of manipulation, tetraacetyl bromoglucose is employed and the product deacetylated.^{470, 994} Phenylthiosides of glucose, xylose, lactose, cellobiose and maltose have been made.¹¹³⁶ β -D-Glucosidothioglycolic acid has been prepared starting with the potassium derivative of ethyl thioglycolate.⁷⁵⁰ Methyl triacetyl- α -L-thioarabinoside has been made from the bromo-compound and potassium methyl mercaptide.^{625.5} Pentaacetyl- β -D-glucopyranose and ethyl mercaptan gave ethyl tetraacetyl-1-thio- β -D-glucopyranoside.^{839.5}

Phenyl tetraacetyl- β -D-thioglucoside,^{162, 470, 994} phenyl triacetyl- β -D-thioxyloside and phenyl triacetyl-D-thioarabinoside have been oxidised by permanganate to the corresponding sulfones.¹⁶²

Phenyl- β -D-glucothioside is changed to levoglucosan by refluxing it with aqueous potassium hydroxide.

The direct combination of cysteine with sugars gives thiazolidine derivatives which are related to glycothiosides. The structure is believed to be:



Some of these compounds are: lactose, m. 130° ; mannose, m. 171° ; galactose, m. 138° ; xylose, m. 133° ; glucose, m. 67° ; and arabinose, m. 153° .¹²⁵⁶

A second class of unsymmetrical sulfides is that of thioglycoses. One compound of this class has been obtained from a natural product. This occurs in yeast in the form of its adenine glycoside.¹³⁴³ The thiosugar is attached to the adenine in position 9.⁴³⁸ The constitution of this sugar was not evident for some time. It is a pentose with one of the hydroxyls replaced by the MeS— group. It can be reduced to the alcohol, $\text{C}_5\text{H}_{11}\text{O}_4\text{SMe}$, m. $115-7^\circ$.¹³⁴² The sugar gives a triacetate. The fact that the sulfur is not removed in osazone formation shows that it is neither in the 1 nor in the 2 position.⁸⁴⁷ The results of oxidation with lead tetraacetate exclude position 3.¹⁴⁴⁵ It was found not to be identical with synthetic 5-thiomethylxylose.^{1147a} Recently the thiopentose has been found to be 5-methylthioribose.¹⁴⁵³ The parent substance is then 5'-methylmercapto-9- β -D-ribofuranosido-adenine.^{67, 1228, 1452} 5-Methylthioribose has been synthesized^{1067, 1227, 1229} and reduced to 5-methylthio-D-ribitol.⁶⁷

Adenylthiomethylribose causes a lowering of the temperature and a slowing down of the basic metabolism in cats and rabbits.⁸¹⁹ Its action is similar to that of other nucleic acid derivatives, lowering the blood pressure in rabbits and relaxing intestine strips.⁴³⁴

Some syntheses have been accomplished in this field. Di(glucosyl-6) sulfide has been prepared from 6-bromoglucose.^{1483a} 3-Thioglucose has been made and converted to the methyl derivative.⁴⁹¹ The 2-methylthio- and 2-ethylthio-glucoses have been produced in several ways, starting with the tetrabenzoates of the mercaptals.²⁰² An acetyl group may be replaced by EtS—. ^{161, 1478} Sulfides are formed by the reaction of methyl and ethyl iodides on the silver salt of thiocellobiose.¹⁴⁸⁴

HYDROXY AROMATIC SULFIDES

Phenol Sulfides

Phenol reacts readily with sulfur di- or mono-chloride, even without a catalyst: ^{406, 1362a, 1362b, 1363a, 1363b}



It is well to write a third reaction:



Sulfur dichloride is in equilibrium with the monochloride and chlorine. Thus the product from it may contain chlorinated compounds as well as disulfides. Sulfur monochloride is a mixture and the product from it may contain monosulfide and trisulfide along with the expected disulfide. Regardless of the starting materials, rather complex mixtures are obtained. As phenol monosulfide crystallizes well, it can be gotten out in pure condition. The isolation of the pure disulfide is much more difficult. In a recent experiment, 52% of the monosulfide was obtained by the reaction of 1 mole of phenol with $\frac{1}{2}$ mole of sulfur dichloride in 1 liter of carbon tetrachloride at -15° . When sulfur monochloride was used under the same conditions, 37% of the monosulfide was isolated from the product.⁹⁴³ It is much the same with alkylated ^{1091, 1191, 1317, 1407} and with halogenated phenols. Sulfur monochloride and *p*-chlorophenol give a mixture of mono- and trisulfide.¹¹⁷⁷ Certain groups may hinder the reaction. Thus methyl salicylate requires a catalyst.⁸²⁰ The product from the sulfurization of a Mannich base from phenol and a diakyl amine has been recommended as an oil additive.^{1050.5}

When the para-position is occupied, substitution takes place ortho to the hydroxyl.¹¹⁷⁷ A statement in one reference is contrary to this.^{1362b, 1363a} In some cases, thionyl chloride gives the same results as sulfur dichloride.^{871, 932b, 1363c}

For the reactions of sulfur chlorides with phenols, carbon tetrachloride is a favorite medium. Elimination of the hydrogen chloride as it is formed is considered beneficial.¹⁰⁹¹ This may be facilitated by carrying out the reaction in a refluxing organic halide.^{973a, 1317}

It has been suggested that the initial product is $S(OPh)_2$, which rearranges: ^{1362a}



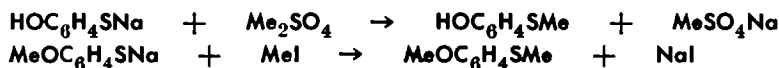
A phenol sulfoxide, $(HOC_6H_4)_2SO$, may be reduced to the sulfide. ^{538, 1293} Excess sulfur may be removed from a crude phenol sulfide by sodium sulfite. ^{13a} Thionyl chloride gives the sulfide with hydroxyacetophenone. ⁷²⁴ Such ketophenol sulfides, in which the alkyl group is large, are claimed as additives to lubricating oils. ⁴³¹

Phenyl ethers ⁸⁷¹ and esters ⁸⁶² undergo the same reactions as phenols but less readily. In the presence of aluminum chloride, phenetol and thionyl chloride give the sulfonium salt, $(EtOC_6H_4)_3SCl$, as the final product. The sulfoxide, $(EtOC_6H_4)_2SO$, which may be the first product, is supposed to react with hydrogen chloride and then with more phenetol. ^{323, 857, 1294}

In patent claims, products obtained by treating substituted phenols with a sulfur chloride are usually mentioned rather than phenol sulfides of specified structure.

Phenol sulfides and their ethers may be made in several other ways. The halogen of an aromatic halide may be activated by a nitro group so that it reacts with sodium sulfide ⁶⁶⁷ or mercaptide. ^{89, 711, 734b} *p,p'*-Diaminodiphenyl sulfide can be converted to the corresponding phenol sulfide by the diazo reaction. ^{803b, 1399} The same has been done with unsymmetrical aminosulfides. ^{89, 179, 670} $[2,5-Me(HO)C_6H_3]_2S$ and the methylolphenyl sulfide, $(o-HOCH_2C_6H_4)_2S$, have been prepared from the diazonium chlorides. ¹¹⁶¹

Unsymmetrical compounds can be obtained from a diazonium chloride and a mercaptide. ^{734a} Hydroxythiophenols and their ethers may be alkylated by conventional methods: ^{49, 532a, 667, 676, 1506a, 1506b}



A lead mercaptide may be used. ⁸⁴⁶ A sodium salt of a thiophenol reacts with an aryl iodide when the two are heated together in the presence of copper: ^{932a, 932b}



The reaction of a sulfene chloride with a phenol has been used frequently: 227, 482, 483, 834, 1502, 1507, 1513, 1515a



A sulfinic acid gives the same result: 286, 290, 443a, 648, 649a, 812



2,4,6-Tris (methylmercapto)phloroglucinol, 1,3,5-(HO)₃-2,4,6-(MeS)₃C₆, has been made by alkylating phloroglucinol with methyl methanesulfonate, MeSO₂SMe.^{54,5}

The ultraviolet^{450b, 1499.5} and infrared¹²⁵³ spectra of several phenol sulfides have been described. The extinction curves of several thioanisoles have been discussed.⁷⁸⁴ The molal refractivities of a hydroxy- and an ethoxy-sulfide have been compared.²⁹³

Phenol Selenides

These are analogous to the phenol sulfides but are much less well known. They can be made by the Grignard reaction^{1351a} or by the diazo, either by replacing an amino group of a selenide by the hydroxyl⁷⁵⁷ or by causing a hydroxybenzenediazonium chloride to react with sodium phenyl selenide.⁷⁵⁶ Selenium oxychloride and phenol give the selenonium chloride, (*p*-HO-C₆H₄)₃-SeCl.¹⁰⁰⁰ Raney nickel removes the selenium from selenides.¹⁴⁷²

Naphthol Sulfides

Naphthol sulfides can be prepared by all of the methods that have been given for phenol sulfides under much the same conditions. β-Naphthol and sulfur dichloride give a mixture which contains the α-monosulfide and also some disulfide.^{9, 632} 4-Chloro-1-naphthol-2 monosulfide is obtained from 4-chloro-α-naphthol and sulfur monochloride.²⁸⁷ β-Naphthol sulfide has been claimed to exist in two isomeric forms.^{649b, 1432} This has caused much discussion.⁸⁴⁵ The methyl ether of β-naphthol gives an almost quantitative yield of the sulfide with sulfur dichloride. This could not be prepared by methylating the naphthol sulfide.^{1042b}

β-Naphthol, heated with sulfur at 170 to 180°, gives an excellent yield of the α-sulfide.^{335c, 1060, 1403} Under the same conditions, an amorphous product is obtained from α-naphthol.^{335c} α-Chloro-β-naphthol, heated with aqueous sodium sulfide gives β,β'-dihy-

droxynaphthyl sulfide.¹¹⁸³ Sulfene chlorides may be used with naphthols as with phenols.¹⁵⁰²

Treating a phenol, or naphthol, sulfide with chlorine or bromine replaces the sulfur by the halogen.^{1042a, 1042b, 1153}

Reactions

The reactions of phenol sulfides are those which are appropriate to phenols and to sulfides. Treating a phenol sulfide with nitric acid converts it to the sulfoxide which is then nitrated.^{667, 709, 1109}

Applications

Various alkylphenol sulfides have been claimed as corrosion inhibitors,^{312a, 313b, 1001, 1130, 1296, 1317} antioxidants^{972a, 973b, 1001, 1286, 1317} and stabilizers for lubricants^{312a, 1189, 1199, 1316a} and for rubber,^{13c, 94, 312a, 1286} and as polymerization inhibitors for vinyl compounds.⁴⁰⁹ Some halogen containing esters of phenol sulfides are antioxidants⁸⁶² and some are useful in extreme-pressure lubricants.^{862, 970c} The tin and antimony salts are said to be stabilizers for rubber.^{13b, 1295} Some alkylphenol sulfides impart desirable characteristics to rubber¹⁴⁷⁵ and some, particularly those containing more than one atom of sulfur, can supply the sulfur necessary for vulcanization.^{139b, 1390, 1474, 1475} Those suitably alkylated may serve as reclaiming agents for natural or synthetic rubber.^{13d, 314, 315, 779.5, 1404.5} Alkylated naphthol sulfides have been claimed for this purpose.⁶³³ Nonionic surface-active agents can be prepared from alkylphenol sulfides and alkene oxides.^{217, 224} Catechol sulfide has been used in making a blue dye.³⁰⁸

Salts of various phenol sulfides are recommended as oil additives,^{13a, 256, 257, 313a, 897, 1296} particularly calcium^{898, 970a, 1190, 1296, 1316b} and barium.^{20a, 313c, 313d, 898, 899, 1190, 1191, 1238, 1316b, 1317, 1407, 1470} A barium compound is said to stabilize lead tetraethyl in gasoline.^{1213.5} The presence of suitable alkyl groups increases their oil-solubility and enhances their value for such uses.¹⁰⁰¹ Aluminum salts have been prepared.^{313c, 977, 1296}

Phenol sulfides may be acetylated by the usual agents.^{34, 313d} Some of the products are said to be useful.^{313d} Compounds of higher molecular weight can be made by coupling two or more molecules of a phenol sulfide with formaldehyde.^{252, 972b, 1296}

There has been considerable interest in phenol sulfides as

germicides^{312b} and pesticides.^{34, 313b} 3-Methyl-4-hydroxydiphenyl sulfide is said to be highly germicidal.²⁸⁶ The same is true of 2,2'-dihydroxy-3,3',5,5'-tetrachlorophenyl sulfide.⁶⁸⁶ The bactericidal properties of a number of substituted-*s*-dihydroxydiphenyl sulfides have been compared.⁴⁰⁶ The activity of hydroxyphenyl alkyl sulfides, $\text{HOC}_6\text{H}_4\text{SR}$, increases with the size of the alkyl group up to butyl.^{976a, 976b} Heavy metal salts of halogenated polyphenol sulfides are effective in the control of the growth of fungi which attack textiles, hides, and leather.^{312c} Acetylated halogenated phenol sulfides are effective fungicides.³⁴

Two sulfide phenols, *p,p'*-dihydroxyphenyl sulfide,²⁷⁷ 1,2-*bis* (*p*-hydroxyphenyl)-1,2-*bis* (methyl-mercapto) ethane¹⁰⁵⁷ and one in which the hydroxyls have been methylated, *bis* (*p*-methoxyphenylethylmethyl) sulfide¹⁷⁷ have shown estrogenic activity.

Halogen Derivatives of Sulfides



α-Halogen Sulfides, RSCH_2X

The simplest group is that of the chloromethyl sulfides, RSCH_2Cl , in which $n = 1$. These resemble the chloromethyl ethers, ROCH_2Cl , in the manner of their formation and in their reactions except those that are peculiar to sulfides. They result from the interaction of formaldehyde, a mercaptan, and hydrogen chloride: 150a, 152, 442



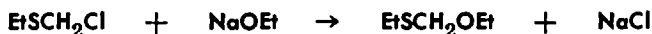
The bromides, RSCH_2Br , are made similarly with hydrobromic acid.¹⁵²

Chloromethyl methyl sulfide, MeSCH_2Cl , can be made by chlorinating methyl sulfide at -20° . Further chlorination gives the di- and trichloro compounds, MeSCHCl_2 and MeSCCl_3 .¹⁵³ Phenyl methyl sulfide has been chlorinated similarly, in three stages, to PhSCH_2Cl , PhSCHCl_2 ,¹⁵³ and PhSCCl_3 .^{153, 1093.5} The chloromethyl benzyl sulfide has been made from the hydroxymethyl benzyl sulfide, $\text{PhCH}_2\text{SCH}_2\text{OH}$, and hydrogen chloride. The hydroxy compound was obtained by heating hydrated formaldehyde and benzyl mercaptan to 130° .^{150.5} *p*-Fluoro-, *p*-chloro-, and *p*-bromophenyl chloromethyl sulfides have been chlorinated to the trichloro stage.^{1093.5}

The chloromethyl sulfides may be regarded as intermediates in the formation of thioformals:



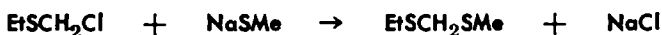
With sodium ethylate a monothioformal is produced: ^{150a}



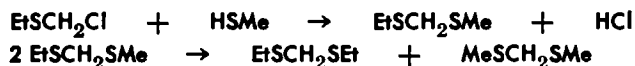
The same compound can be made, the other way around, from a chloromethyl ether.^{144b, 1455b} A monothioformal disproportionates in the presence of hydrogen chloride:



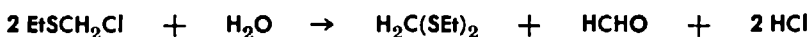
Mixed thioformals can be obtained from a sodium mercaptide: ¹⁵⁴



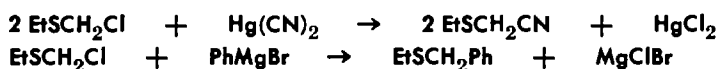
The mixed thioformal is formed also in the absence of alkali, but the hydrochloric acid so produced catalyzes the rearrangement:



With water a sort of disproportionation follows partial hydrolysis: ^{150a}



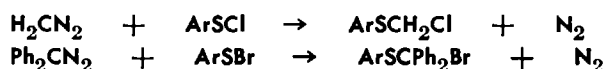
A cyanide is formed with a metal cyanide and a sulfide with a Grignard reagent: ^{150a}



Perbenzoic acid oxidises to the sulfone, $\text{EtSO}_2\text{CH}_2\text{Cl}$, $b_{14} 128^\circ$, $m. 33^\circ$.^{150a} The benzyl compound is oxidised by peracetanhydride to the sulfone.^{168a} In chloroform solution the oxidation by ozone stops at the sulfoxide, $b_{0.2} 70^\circ$.^{151a}

Quite a number of the chlorides, RSCH_2Cl , have been prepared and caused to react with sodium alkylmalonic esters^{153.5} and the resulting products, $\text{RSCH}_2(\text{R}')\text{C}(\text{CO}_2\text{Et})_2$, have been converted to barbiturates, $\text{RSCH}_2(\text{R}')\text{C}(\text{CONH})_2\text{CO}$.^{1428, 1429, 1430a, 1430b} The chlorides, RSCH_2Cl , react with alcohols and with carboxylic acids with the elimination of hydrogen chloride.^{1302b}

Diazomethane and its diphenyl derivative react with aryl sulfenyl halides to give compounds of this type: ¹²⁴⁷



When the triphenyl compound, PhSCPh_2Cl , is shaken with mercury, it is decomposed into tetraphenylethylene and benzophenone mercaptole.¹²⁴⁶

Methyl sulfide and chloromethyl methyl sulfide form the sulfonium chloride, $\text{Me}_2(\text{MeSCH}_2)\text{SCl}$.^{153.7} Valuable products are said to be formed by the condensation of a chloromethyl sulfide with an alpha or gamma amino acid in the presence of an acid binding agent.^{694c} Aryl trichloromethyl sulfides, ArSCCl_3 , have been compared with other insecticides and fungicides.¹⁰⁹³

Higher aldehydes react similarly, but have not been exploited extensively. Acetaldehyde gives the α -methyl compound.^{152, 732a}



A number of these, like their lower homologs, have been used in making barbiturates.^{1428, 1429, 1430a, 1430b}

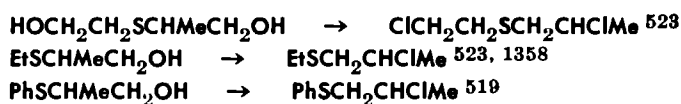
Analogous selenium compounds are known.¹⁴⁶⁵

Chloromethyl sulfides will be discussed again in the chapter on mercaptals in volume III.

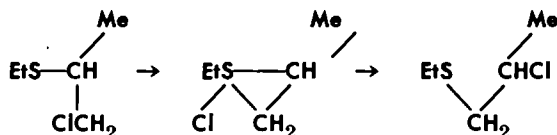
Other Halosulfides

The remarkable properties of β, β' -dichloroethyl sulfide aroused much interest in the study of other chlorinated sulfides. Much attention has been given to the monochlorides, $\text{RS}(\text{CH}_2)_n\text{Cl}$ and $\text{ArS}(\text{CH}_2)_n\text{Cl}$, of which many have been prepared, both aliphatic and aromatic. Besides the straight-chain compounds, with values of n from 1 to 18, there have been various branched chains also. The general result is that the peculiar properties of mustard gas are manifested only when the chlorine is in the beta position to the sulfur atom. All of these chlorosulfides in which $n = 2$ are more or less vesicant.^{220, 350b, 567a, 574, 780a, 780b, 956b, 1023, 1298}

It is unnecessary to go into details of the preparation of the chlorosulfides. Almost all of the hydroxysulfides enumerated in the earlier sections have been converted into them, usually by means of thionyl chloride. In certain cases, the reaction of a hydroxysulfide, with either thionyl chloride or hydrochloric acid, leads to unexpected results; the RS-group shifts to the α -carbon and the chlorine is found on the β -carbon:

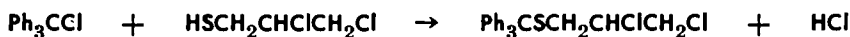


This is explained by assuming the formation of a sulfonium intermediate, once the hydroxyl is replaced by chlorine:



As will be seen later on, this is of importance in elucidating the chemistry of mustard gas.

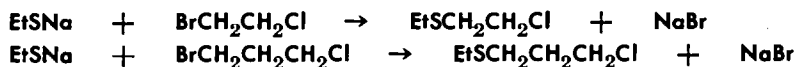
Only in special cases can a chlorosulfide be prepared from a chloromercaptan and an alkyl halide:



This takes place when the reactants are simply heated together.^{344b} Under these conditions, the tertiary halogen is more reactive than either of the others. In the presence of alkali the reverse would be true. A moderate yield of a β -chlorosulfide has been obtained by alkylating a mercaptan with β -chloroethyl *p*-toluenesulfonate.⁵⁵⁴

Furfuryl mercaptan, treated with a Grignard reagent, forms $\text{C}_4\text{H}_3\text{OCH}_2\text{SMgBr}$ which has been made to react with the *p*-toluenesulfonic esters $\text{MeC}_6\text{H}_4\text{SO}_3\text{CH}_2\text{CH}_2\text{Cl}$ and $\text{MeC}_6\text{H}_4\text{SO}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Cl}$.⁵⁵⁹

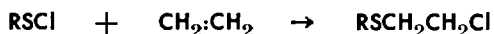
Advantage may be taken of the difference in reactivity of the halogens in mixed dihalides. Ethylene and trimethylene chlorobromides have been caused to react with mercaptides:⁸¹⁰



The addition of mercaptans to tetrafluoroethylene,^{407, 602, 794, 1146} to trifluorochloroethylene,^{794, 1146} and to 1,1-difluoro-2,2-dichloroethylene¹¹⁴⁶ gives β -chloroethyl sulfides. Methyl,⁷⁹⁴ ethyl,^{407, 602, 794, 1146} *i*-propyl,⁷⁹⁴ butyl, hydroxyethyl,¹¹⁴⁶ and phenyl⁷⁹⁴ mercaptans have been used in such additions. Methyl, ethyl, and hydroxyethyl mercaptans have been added to perfluoropropylene.^{794.5} β -Chloroethyl mercaptan has been added to unsaturates.^{1047a}

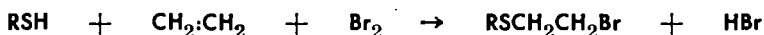
The addition of a sulfene chloride to an unsaturate gives a

β -halogen sulfide: 167, 202.5, 324.5, 715, 765, 766, 767, 768b, 768.5, 834, 1062, 1237



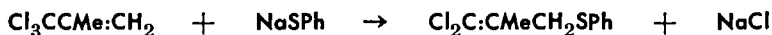
Perchloromercaptan has been added to octene-1,⁷⁸⁵ PhSCl and 2,4-Cl(O₂N)C₆H₃SCl to cyclohexene,^{167, 1401, 1473} to cyclopentene, and to isobutene, and *p*-O₂NC₆H₄SCl to cyclopentene and cyclohexene.¹⁴⁰¹

Possibly connected with this is the formation of RSCH₂CH₂Br by passing ethylene into a carbon tetrachloride solution of the mercaptan and bromine at -20°: ¹²³⁷



This is one of the reactions of sulfene chlorides which have been considered in Chapter 3, Volume I. It will come up again in the next chapter when the formation of mustard gas is considered.

The reaction of thiophenol with 3,3-dichloropropylene is partly addition with loss of hydrogen chloride and partly mercaptal formation. The products are PhSCH₂CH:CHCl and H₂C:-CHCH(SPh)₂.^{356, 357} The reaction of sodium thiophenate with trichloro-*i*-butylene is of the second kind: ³⁵⁷



There are a few examples of the preparation of halosulfides by direct halogenation. Methyl ethyl sulfide is chlorinated to MeSCHClCH₃.¹⁵³ Allyl sulfide has been brominated with N-bromosuccinimide.⁵⁷ The halogen enters the α -position.

The vapor pressures of a number of β -chlorosulfides have been measured from 0 to 60° and equations derived by which they can be calculated for any temperature.¹¹⁴⁸

Reactions

There are tremendous differences in the rates of hydrolysis of RS(CH₂)_{*n*}Cl when *n* has different values. The maximum is at *n* = 2; BuSCH₂CH₂Cl is hydrolyzed instantly at 80° while PhSCH₂CH₂CH₂Cl and PrSCH₂CH₂CH₂Cl are only 60% hydrolyzed in 7 hours at 150°. ^{106b, 112c} Mustard gas hydrolyzes 400 times as fast as dichloroethyl ether. ^{986b} β -Chlorosulfides hydrolyze 10,000 times as fast as the corresponding chloroethers. ^{150c, 156} However, the hydrolysis of EtSCH₂Cl and of (ClCH₂)₂S is

much slower than that of the corresponding oxygen compounds.^{150b, 150c} The relative rates of ring closure of the halides, $\text{Rs}(\text{CH}_2)_n\text{Cl}$, to sulfonium compounds are comparable to those of hydrolysis.^{108, 110, 113} This will be taken up in the chapter on cyclic sulfides in Volume III.

The reactivity of the sulfur atom, as measured by its rate of oxidation by hydrogen peroxide, is affected by the number and position of the halogens. The absorption spectra and other properties of the compounds are also influenced.⁷¹²

A chlorine atom in the β -position labilizes the sulfide linked to an aryl group. When 2-chloroethyl *p*-tolyl sulfide is treated with potassium cyanide, the expected nitrile is not found. In its stead *p*-tolyl mercaptide $\text{MeC}_6\text{H}_4\text{SK}$ is formed and reacts with the starting material to produce the bis-sulfide, $\text{MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{SC}_6\text{H}_4\text{Me}$. Refluxed with dimethylaniline the 2-chloroethyl *p*-tolyl sulfide is cleaved and *p*-tolyl mercaptan is produced. 2-Chloroethyl butyl sulfide, $\text{BuSCH}_2\text{CH}_2\text{Cl}$, is not cleaved in this way.⁸⁸⁶

The effect of substituents in the phenyl group of $\text{PhSCH}_2\text{CH}_2\text{Cl}$ on the reactivity of the halogen has been studied.^{66b, 106b}

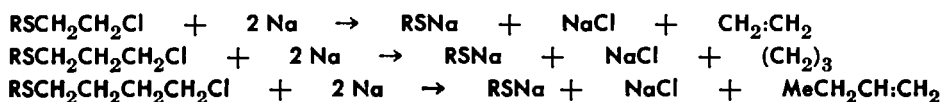
The $-\text{SO}_2-$ group has much the same effect as the $-\text{S}-$ atom; $\text{EtSO}_2\text{CH}_2\text{CH}_2\text{Br}$ is immensely more reactive than $\text{EtSO}_2\text{CHBrMe}$.¹¹⁴⁴

Comparisons of the rates of reaction with potassium iodide in alcohol tell a different story. Taking the rate with butyl chloride as 1, the figures are:⁷⁸¹

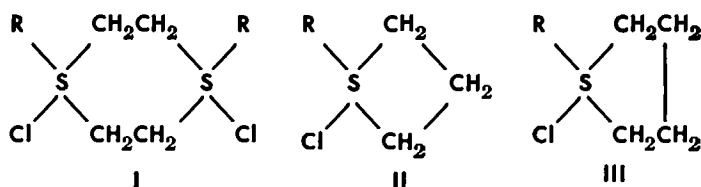
$\text{MeSCH}_2\text{CH}_2\text{Cl}$	1.52	$\text{MeSCH}_2\text{CH}_2\text{CH}_2\text{Cl}$	2.52	ratio	1.7
$\text{PhSCH}_2\text{CH}_2\text{Cl}$	0.56	$\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{Cl}$	2.74		4.9
$\text{PhOCH}_2\text{CH}_2\text{Cl}$	0.30	$\text{PhOCH}_2\text{CH}_2\text{CH}_2\text{Cl}$	1.67		5.6

The sulfur compounds react somewhat faster than the corresponding oxygen. In all cases the chlorine is more reactive when it is in the gamma position than in the beta.^{106a, 107, 780a} The reactivity of ClCH_2SEt with potassium iodide in alcohol has been measured.¹⁰¹⁶

In their reactions with sodium, the chlorosulfides, $\text{RS}(\text{CH}_2)_n\text{Cl}$, differ markedly from each other according to whether n is 2, 3 or 4.



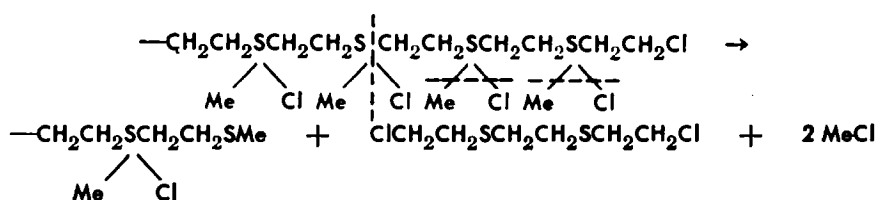
When n is 3, a high yield of cyclopropane is obtained but when n is 2 or 4 there is no cyclic hydrocarbon.⁶⁸⁹ Possible sulfonium intermediates are:



It is well known that a cyclopropane ring is more likely to form than a cyclobutane.

Magnesium does not react with $\text{PhSCH}_2\text{CH}_2\text{Cl}$ but does with $\text{PhSCH}_2\text{CH}_2\text{Br}$ which it converts to phenyl mercaptan, ethylene and $\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{SPh}$.^{21a}

As methyl β -chloroethyl sulfide is both an alkyl halide and a sulfide, there is the possibility of its forming a polysulfonium compound. In one synthesis in which it was employed, a by-product was isolated which must have come from the dichloride, $\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl}$. This was accounted for by assuming the formation and breakdown of such a sulfonium compound:¹²⁹⁹



In an effort to arrive at an explanation of the vesicant action of β -halosulfides, their reactions with amines,^{350a} aminoacids,¹⁴¹⁴ and proteins^{259, 1326, 1328} have been studied. β -Chloroethyl butyl sulfide, $\text{BuSCH}_2\text{CH}_2\text{Cl}$,^{1328, 1480} and β -chloroethyl benzyl sulfide, $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{Cl}$,^{771, 1480} containing radioactive sulfur have been employed in following such reactions.^{1328, 1480} This will be treated again in connection with mustard gas.

Compounds such as $\text{ArOCH}_2\text{CH}_2\text{SR}$, in which Ar is a wax-phenol and R is a long-chain alkyl group, are said to be pour-point depressants. They are made from the chlorides $\text{RSCH}_2\text{CH}_2\text{Cl}$.^{970b}

The dehydrohalogenation of a β -chlorosulfide by a sodium alcoholate gives a vinyl sulfide:^{211, 1125}



Halogenated Aromatic Sulfides

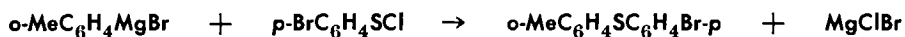
These have been known since 1874.^{803b} In recent years, a number have been prepared in the search for compounds efficient as pesticides, either alone or in combination with natural products.⁵⁴⁰

p,p'-Dichlorophenyl sulfide can be prepared by the chlorination of phenyl sulfide.^{149, 803b} It is probable that the first reaction is the addition of the chlorine to the sulfur atom to form Ph_2SCl_2 and that the chlorine then migrates to the ring. Further addition and migration may take place.^{386, 500b} The same compound is one of the products when benzene is treated with sulfur chloride.¹⁴⁸ It is formed by treating diphenyl sulfoxide, Ph_2SO , with phosphorus pentachloride^{961, 1329} or with thionyl chloride and aluminum chloride.⁸⁷¹ Methyl phenyl sulfide, formaldehyde, and hydrochloric acid give 2,4- $(\text{ClCH}_2)_2\text{C}_6\text{H}_3\text{SMe}$.¹⁴⁷⁹

The story of the bromine derivatives of phenyl sulfide is practically the same as that which has just been told about the chlorine. The bromine addition product is formed at a low temperature, but is unstable. Even below 0° the bromine migrates:¹⁴⁷



The final product may contain some of the mono-^{147, 149, 176, 177, 500b} as well as the dibromosulfide, $(p\text{-BrC}_6\text{H}_4)_2\text{S}$.^{147, 149, 176, 177, 386, 500b, 803b, 1201, 1486} This can be obtained through the diazonium salt from the corresponding *p,p'*-diaminophenyl sulfide.^{803b, 1363d} An unsymmetrical compound has been made:¹³⁹⁶



The bromination of methyl phenyl sulfide follows the same course as the diphenyl:^{177, 1508}

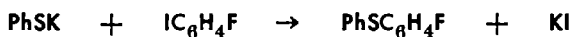


Substituents in the phenyl group have their characteristic effects on the course of the bromination.^{291, 676, 1511} Nitro groups may prevent the addition of bromine to the sulfur and its entrance into the ring.⁵⁰¹ In the case of *m*-dibenzylmercaptobenzene, the bromine enters the 4 and 6 positions⁴⁶⁸ and the 1,5-positions in 6-methylmercapto- β -naphthol.¹⁵⁰⁵ The same compounds may be made by the diazo reaction.¹⁸⁹ A bromophenyl sulfide can be

made, the other way around, by alkylating a bromophenyl mercaptan, such as 4-bromodithiocatechol.⁵⁹²

Little need be said about iodophenyl sulfides. Ethyl *p*-iodophenyl sulfide^{992, 1460} and *p,p'*-diiodophenyl sulfide^{803b} have been made from the corresponding aminosulfides by the diazo reaction. The ethers *p*-ROC₆H₄SC₆H₄I-*p*, have been obtained from the amino compound similarly.^{734b}

p-Fluorophenyl sulfide has been prepared from *p*-iodophenyl fluoride:



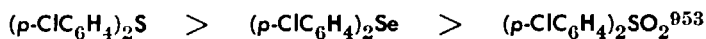
p,p'-Difluorophenyl sulfide has been obtained by reducing the sulfoxide which was made by the reaction of thionyl chloride on fluorobenzene in the presence of aluminum chloride.⁸⁴²

The benzyl sulfides, (*p*-ClC₆H₄CH₂)₂S,⁷¹⁹ (*p*-BrC₆H₄CH₂)₂S,⁷¹⁸ and (2,5,3,6,4-Me₂Br₂(HO)C₆CH₂)₂S⁵⁰ are readily obtained from the corresponding benzyl bromides and alkali sulfide. A study has been made of the kinetics of the oxidation of (*p*-ClC₆H₄CH₂)₂S by hydrogen peroxide^{1066b} and by peroxybenzoic and *para* substituted peroxybenzoic acids^{1066a} to the sulfoxide.

Under certain conditions, the sulfur of a halogenated phenyl sulfide may be removed by treatment with chlorine¹¹⁵³ or with sulfur.¹³³

Polarizations have been measured and dipole moments calculated for Ph₂S, *p*-ClC₆H₄SPh, and (*p*-ClC₆H₄)₂S.⁶⁰⁰ The dipole moments of the ortho, meta, and para chlorophenyl phenyl sulfides, ClC₆H₄SPh, have been determined.¹¹⁹⁵ Ultraviolet spectra of several halogenated phenyl sulfides have been recorded.⁹⁰⁷ The crystal structure of the dibromosulfide has been determined.^{1394b} The valence angle of the sulfur is 109.5°. ^{1394a} The chlorine and bromine derivatives, (*p*-ClC₆H₄)₂S and (*p*-BrC₆H₄)₂S, form a continuous series of mixed crystals.¹¹⁷¹ The sulfides do not form mixed crystals with the sulfoxides or sulfones.¹¹⁶⁹

As pesticides, the order of activity is:



p,p'-Dichlorodiphenyl sulfide is synergistic with nicotine^{936, 937, 938} and with DDT.⁹¹⁵ An extensive study has been made of the fungistatic and bacteriostatic properties of halogen and other

derivatives of diphenyl sulfide.¹⁰⁹⁴ Methyl and ethyl pentachlorophenyl sulfides have been tested.^{1351,5} 2,2',4,4'-Tetrachlorodiphenyl sulfide has been tested, along with many other compounds, as a growth stimulant.^{1036, 1379} The dibromosulfide has been tested as a synergist for nicotine.⁹³⁸

The methods for preparing halogenated phenyl selenides are similar to those for the sulfides. The halogen of diphenyl selenium bromide,^{269, 417} or chloride,⁸⁰⁴ migrates to the ring:



This compound can be obtained from benzene, selenium dioxide, and aluminum chloride.⁸⁸² *o*-Chlorobenzyl selenide, $(\text{o-ClC}_6\text{H}_4\text{CH}_2)_2\text{Se}$, has been made from *o*-chlorobenzyl chloride and sodium selenide.¹³⁰⁸

CHAPTER 5.

Mustard Gas



Introduction

Since the invention of gunpowder, there has been no more important change in the conduct of land warfare than that brought about by the introduction of gases in World War I, and of all the agents used, mustard gas overshadows all the rest. Had a large supply been available and had this been used over the whole front in the first attack, the war might have been won by the Germans, almost in a day. It was a new and unanticipated weapon against which the Allies had no protection and which they were, for a short but terrifying time, unable to produce.

Aside from its military use, mustard gas is most interesting, and has occasioned a multitude of investigations, both chemical and physiological. A sulfur atom in a molecule has a decided influence on the reactivity of atoms or groups, particularly those on carbon atoms in the beta position. This is most strikingly illustrated in β,β' -dichloroethyl sulfide, commonly known as mustard gas. The use of this compound during World War I has led to its intensive study which has been extended to related compounds. The subject has been reviewed by Jackson⁷²¹ * and

* As most of the authors quoted in this chapter have contributed also to Chapters 4, 6, and 7, all of the references for the four chapters are assembled in the Bibliography placed at the end of Chapter 7.

discussed in many articles.^{1, 29, 130, 225, 266, 382, 498, 636, 671a, 770, 948, 958, 1079, 1088, 1138, 1254b, 1278, 1421} Its uses and its chemistry are described in books on chemical warfare.^{27, 45, 143, 365, 423, 446, 476, 548, 596, 605, 621, 714, 716, 854, 856, 863, 954, 955, 1003, 1015, 1226, 1404, 1411, 1434, 1448}

Historical

In 1822, "in examining the action of ethylene on the chlorides of sulfur and iodine Despretz³⁶⁹ observed the formation of products worthy of remark," but the information is too meager to show just what he had. Regnault¹¹⁵⁴ and Riche¹¹⁷⁴ chlorinated ethyl sulfide, but their products do not seem to have been vesicant.

Niemann¹⁰⁴⁰ passed dry ethylene into sulfur chloride and tried to purify the product by distillation, but ran into difficulty. Analysis corresponded to $C_4H_8S_2Cl_2$:



Had he distilled the material at a low pressure he should have isolated the pure compound $C_4H_8S_2Cl_2$. He describes his product as raising blisters and being dangerous to handle.

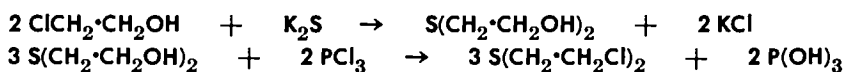
Guthrie⁵⁹⁴ made an extensive study of the reaction of ethylene with sulfur chloride but did not isolate the pure dichloroethyl sulfide. It must have been produced but was largely destroyed by the treatment he gave it. His product was vesicant, but evidently not comparable to pure mustard gas. He mentions a headache after swallowing three or four drops of it.

Guthrie's product is listed by Beilstein,⁹⁸ 4th Ed., as the disulfide, $(ClCH_2CH_2)_2S_2$, specific gravity 1.346 at 19°, and not connected with the monosulfide of Victor Meyer. The fact that chlorination produced a derivative of the monosulfide, $(C_2H_2Cl_3)_2S$, was mentioned but its significance was not pointed out. It remained for the English chemists in 1917 to discover that the product made by Despretz, Niemann, and Guthrie was actually mustard gas.

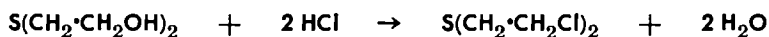
The history of β, β' -dichloroethyl sulfide as a pure compound begins with Victor Meyer.^{956a, 956c} He hoped to get a cyclic pentamethylene sulfide derivative from it and malonic ester:



This objective was not reached, but the intermediate, $S(CH_2 \cdot CH_2Cl)_2$, attracted attention on account of its intense vesicant action. He made the intermediate in two steps:



The thiodiglycol was isolated and its hydroxyls replaced in a clean-cut reaction which left no doubt as to the structure of the final product. Its boiling and melting points were determined. Clarke^{292a} found that the hydroxyls could be replaced more simply by warming the thiodiglycol with concentrated hydrochloric acid:



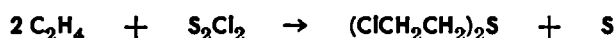
The β,β' -dichloroethyl sulfide was placed in its proper niche in Beilstein and rested there undisturbed for many years; its formula suggested interesting syntheses, but its extreme toxicity deterred chemists from using it.

Chlorine was introduced by the Germans at Ypres April 22, 1915 and was effective against troops lacking any sort of protection. Phosgene and other gases followed, but all of these were lung injurants and were harmless to those protected with masks. The development of masks and absorbent charcoal kept pace with the introduction of new gases of this kind. Sometime in 1917, the Germans began the manufacture of mustard gas according to the Victor Meyer-Clarke method and launched an attack with it, again at Ypres, on the night of July 12, 1917. This came as a complete surprise to the Allies and caused thousands of casualties. It was soon identified by the British¹¹¹⁹ who began an intensive search for methods of manufacture. Ethylene chlorhydrin which was required for the Victor Meyer method was generally known, but only in Germany had its large-scale manufacture been worked out. The British dug up the articles by Niemann and Guthrie and showed that the ethylene-sulfur chloride reaction produces the monosulfide, $\text{S}(\text{CH}_2\text{CH}_2\text{Cl})_2$. Knowing from Victor Meyer's work just what to look for, they were able to isolate it. This was cabled to Washington on January 16, 1918 and found the American chemists already at work along the same line. The laboratory results were quickly translated into large-scale plant operations. There has been considerable controversy^{584, 1111a, 1447, 1463} as to the allocation of the credit for this development which went on simultaneously on both sides of the ocean. Only one who has carried a chemical process from the laboratory through semi-works to large-scale operation can appreciate the difficulties that had to be overcome.

In this case, these were made ten times greater by the extreme toxicity of the product and by having to put through the development while the fate of nations hung in the balance. Guthrie and Niemann had showed that ethylene can be taken up by sulfur monochloride, but many laboratory experiments had to be carried out in order to find the proper conditions for obtaining a satisfactory yield of the right product. Considering the difficulties that had to be surmounted and the importance of the achievement, there is plenty of credit to go around, not only for those named before, but for scores of others who toiled long hours always in mortal danger.

In the United States,³⁸⁵ the laboratory investigation was carried on at the American University in Washington. A small-scale experimental plant was built in Cleveland, Ohio, where the findings of the laboratory were put through the semi-works stage and designs made for large-scale reactors which were set up at Edgwood, Maryland, and at several cooperating commercial plants.

A difference of 30° in the temperature at which the ethylene is absorbed by the sulfur chloride would not seem to be a matter of much importance, but actually it is. In England and in this country the reaction was carried on for some months at 60°. The equation is commonly written:



According to this the sulfur that is produced is 16% of the product. Actually about half of this amount did separate on standing. Extensive experiments were made on the purification of the crude mustard gas. Vacuum distillation gave a product 98% pure, but on a large scale there was considerable decomposition due to the long heating. Flash distillation by flowing the crude material down a 2-in. steam-jacketed iron pipe 10 ft. long inclined at an angle of 30° under a 29-in. vacuum gave a product about 94% pure. The capacity of such a still was about 1 ton of condensate per tube per 24 hours. As the time of contact with the iron was short the decomposition was negligible.

In August 1918, information came from England about the 30°C. process which is specifically known as the Levinstein process. It had been found that very pure ethylene is absorbed rapidly by sulfur monochloride at as low as 30°C in the presence

of crude mustard gas. Into a reactor partly filled with the product from a former run pure ethylene is passed while sulfur monochloride is fed in. It is important to maintain the proper concentration of sulfur chloride.⁴⁵⁸ When the reactor is nearly full the sulfur chloride feed is cut off to allow complete saturation with the ethylene. The major part of the product is drawn off and the cycle of operations is repeated. As far as the reactor is concerned the operation is practically continuous. The peculiarity of the process is that the sulfur called for by the previously given equation does not separate out, either in the reactor or on long standing. This is a great convenience both in manufacturing and in shell filling. Had the separation of sulfur from the 60° product always taken place at the same stage of the process in exactly the same way and had the sulfur come down in nice granular crystals, it would not have been so bad, but this was not the case. Much grief was caused by the deposition of sulfur in pipe lines. To this day no one knows exactly what is in the "Levinstein" mustard but, curiously enough, physiological tests disclose no appreciable difference between it and the highly purified material. It was adopted as standard by the Allies. On November 1, 1918 the capacity at Edgwood was 30 tons per day, which was to have been raised to 80 in December with 75 tons per day from two other plants. The American achievements were chronicled by Herty.⁶⁴³ Pope^{111b} claimed that by the end of the war, the Allies' production was 30 times that of the Germans and at 1/30 of the cost per pound. The French^{566, 748, 930} used the same method. German factories and methods were inspected by Norris¹⁰⁴⁵ and the manufacture described by Carr.²⁸⁰

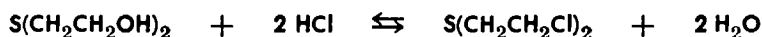
The thiodiglycol process⁵⁷⁷ has been investigated in the United States, but never carried to large-scale operation.

Preparation

FROM THIODIGLYCOL

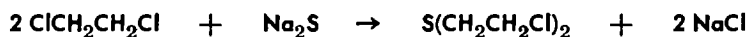
As mentioned before, the first preparation of pure β,β' -dichloroethyl sulfide was by Victor Meyer^{95b} from thiodiglycol by the action of phosphorus trichloride. Instead of this thionyl chloride¹³²³ and phosgene²⁷⁵ have been recommended. It was found by Clarke^{292a} that the hydroxyls of thiodiglycol are so reactive that concentrated hydrochloric acid serves just as well. The thiodiglycol is mixed with concentrated hydrochloric acid and

warmed at 60 to 75°. The mustard gas separates as a heavy layer and is drawn off.⁵⁷⁷ As the reaction is reversible, it is customary to pass hydrogen chloride into the mixture:



A quick method is to mix 25 cc. of thiodiglycol with 75 cc. of concentrated hydrochloric acid and warm to 60° for 30 minutes.¹¹⁶ Or 122 g. (1 mole) "Kromfax solvent" and 830 cc. concentrated hydrochloric acid are mixed and heated at 80 to 90° for 1 hour. The lower layer is separated and freed from hydrochloric acid by passing dry air through it. The yield is 94% of 96% purity, melting at 13°. The pure compound may be obtained by distillation, b_5 81°, b_{10} 93°.¹¹⁵¹ An apparatus for the laboratory preparation has been described.⁸⁶¹ The preparation of mustard gas containing radioactive sulfur and deuterium has been described.^{182b} For synthetic use, it is more convenient and safer to make the mustard gas when and where it is to be used than to procure it. It is expensive to ship and dangerous to store. The apparatus should be so arranged that it is generated and handled in a closed system.

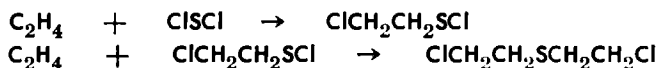
Attempts have been made to stop the reaction between an ethylene dihalide and sodium sulfide at the halfway stage:³⁶⁰



In one experiment of this kind,¹¹⁵⁸ a burn was produced by the liquid immediately after the reagents were mixed. As soon as one of the halogens of ethylene chloride reacts, the other finds itself in the beta position relative to a sulfur atom. This so intensifies its reactivity that the halogen of the ethylene chloride cannot compete with it. An interesting experiment would be to react an excess of decamethylene chloride with sodium sulfide. In such a case it may be possible to isolate some of the intermediate, $\text{S}(\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{Cl})_2$.

FROM SULFUR CHLORIDES AND ETHYLENE

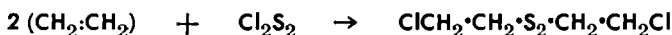
Two simple reactions can be written:



Mustard gas has been obtained by passing ethylene into sulfur dichloride in the presence of absorbent charcoal at 40 to 45°.⁵⁴⁶

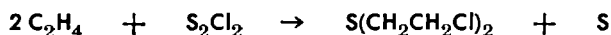
and by spraying the dichloride into ethylene and catching the product on a cold surface.¹⁰¹⁹ The best results were with a mixture of 75% of SCl_2 and 25% of S_2Cl_2 . The reaction is difficult to control. Sulfur dichloride readily parts with chlorine and mustard gas is easily chlorinated; the product is liable to contain a considerable amount of chlorinated mustard. There is no record of this method having been used on a large scale. The three methods of preparation have been compared.³⁵⁹

The reaction of ethylene with sulfur monochloride has received much attention and many equations have been written. It is probable that most of them represent reactions that do go on in the complex mixture, but it is certain that no one or two of them tell the whole truth. Spring and Lecrenier¹³¹⁰ wrote:



They proved this to their satisfaction by oxidising Guthrie's product to the sulfonic acid, $\text{ClCH}_2\text{CH}_2\text{SO}_3\text{H}$, from which they made taurine, $\text{NH}_2\text{CH}_2\text{:CH}_2\text{SO}_3\text{H}$. It is now known that the crude product contains polysulfides, the oxidation of which gives chloroethanesulfonic acid. Gibson and Pope⁵⁴⁶ are inclined to the same view of the reaction at low temperature, but say that when the absorption is carried on at 60° , or above, and the product heated to 100° the sulfur separates out leaving 90 to 98% of mustard containing 3% of sulfur.¹¹¹² If the temperature is kept at 60° , the deposition of the sulfur is slow. Alcohol increases the yield up to 98%. At 30, 60, and 100° the relative rates of the reaction are 1, 2.5, and 7 and the corresponding yields are 70, 80, and 90%. The trisulfide, $(\text{ClCH}_2\text{CH}_2)_2\text{S}_3$, b_p 146.5° , m . 27° ,⁹¹² was isolated.

Two investigators suggested the formula, $(\text{ClCH}_2\text{CH}_2)_2\text{S:S}$, assuming that the ethylene reacts with sulfur chloride in its unsymmetrical form, $\text{Cl}_2\text{S:S}$. The molecular weight found, 187, is close to 191, the calculated. Against this formula is the fact that no authenticated disulfides, $\text{R}_2\text{S:S}$, are known.^{584, 1024} According to the equation, 16.76% of the product should be sulfur:



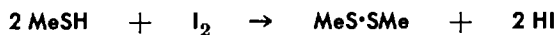
Some sulfur, but usually less than half of this amount, separates out from the product obtained at 60° ⁴⁵⁷ and ammonia causes further precipitation. When the reaction is carried out at 30° , as in the Levinstein process, little or no sulfur comes out, but moist

ammonia may throw down 40 to 45% of the calculated. The melting point of the crude mustard is not changed by this precipitation. This led to the supposition that this amount of the sulfur is in the colloidal state.^{457, 917} Bennett concluded that the product is a two-phase system in which the continuous phase is nearly pure $S(CH_2CH_2Cl)_2$ and the disperse phase a liquid, chiefly sulfur. The relative viscosity is characteristic of a liquid-liquid two-phase system. For comparison, he prepared the disulfide $ClCH_2CH_2S\cdot SCH_2CH_2Cl$, starting with monothioethylene glycol, $HSCH_2CH_2OH$. The disulfide boils at 155° at 30 mm. and has $d_{20/4} 1.3375$. Its vesicant action is one third that of the monosulfide, far less than that of Levinstein mustard.^{104a}

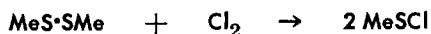
The curious fact that the separation of a part of the sulfur does not change the melting point has been explained satisfactorily by recent findings which are given here. The nonmustard portion is a mixture of polysulfides. If 100 molecules of a pentasulfide lose sulfur, which precipitates out, to go down to 100 molecules of a trisulfide, the number of molecules of the polysulfide in solution is not changed and the melting-point depression remains the same. If the sulfur remains in combination, the average molecular weight of the mustard and polysulfides must be 191, regardless of what polysulfides are present.

The Formation of Levinstein Mustard

In approaching this subject one must remember the organic chemist's dictum that all possible reactions will take place, the velocity of each being determined by the concentrations of the reactants and by a specific rate constant. However, one must forget about the fixedness of bonds on which the organic chemist's ideas of structure are built. Unlike $C-H$, $C-O$, $C-S$ and $C-Cl$ bonds, $S-H$, $S-Cl$ and $S-S$ bonds are labile. Structures which involve these labile bonds do not have the validity of our usual formulae. Thus methyl mercaptan, which is not ionized, reacts almost as quickly as if it were. Methyl disulfide falls apart easily. The mercaptan can be titrated with iodine:



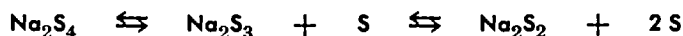
When the calculated amount of chlorine is passed into a solution of methyl disulfide at -20° it is cleaved:



This reacts instantly with potassium iodide: ²⁰³



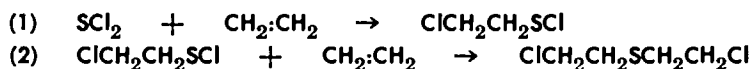
Sodium polysulfides are in labile equilibrium with each other and with free sulfur:



In an actual experiment, sodium tetrasulfide in aqueous solution was reduced to the disulfide by repeated extraction with carbon disulfide. The amount of sulfur removed was 98.8% of the calculated. The reaction of dichloroethyl ether with sodium tetrasulfide in aqueous solution gives 16% of thioxane, showing the presence of sodium monosulfide even in the tetrasulfide.³⁴⁵

The Levinstein Reactions

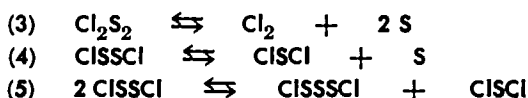
As was shown by Conant and coworkers ³¹⁰ in 1920 and recently verified by Fuson and associates,^{518, 522} the fundamental reactions are the addition of sulfur dichloride to ethylene to form 2-chloroethylsulfenyl chloride and the addition of this to a second molecule of ethylene:



The sulfenyl chloride was prepared, though not in pure form, by the earlier workers.^{310, 911a} They passed a limited amount of ethylene into cold, well-stirred sulfur dichloride. Chlorinolysis of 2,2'-dichloroethyl disulfide gives a much purer product:



This is an orange-colored liquid, b_{15} 47 to 47.5°, $n_{20/D}$ 1.5290. It combines with ethylene to give mustard gas. With cyclohexene, the product is 2-chloroethyl 2-chlorocyclohexyl sulfide⁵²² and with propylene, it is $\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{CHClCH}_3$.⁵²³ Reactions (1) and (2) have been well established but they require sulfur dichloride, while actually the monochloride is used. There is no doubt that the sulfur chlorides are in labile equilibrium with each other and with sulfur and chlorine:



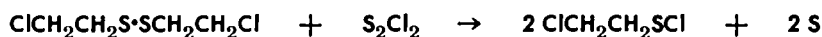
By the reaction of sulfur monochloride, in excess, on hydrogen disulfide, oils have been obtained containing up to twenty-four atoms of sulfur to two atoms of chlorine. Raman spectra indicate long chains of sulfur atoms.^{448, 449} Chlorine tetra- and pentasulfides may exist.

The presence of 2-chloroethyl 2-chlorovinyl sulfide, a chlorination product of the monosulfide, in Levinstein mustard gas is evidence for reaction (3). Equilibrium (4) which has been considered a source of the dichloride,³¹⁰ accounts for the monochloride acting as a sulfurizing agent. Equilibrium (5) may furnish dichloride and account for the presence of large amounts of trisulfides in the nonmustard portion of the crude.⁵¹⁸ The fact that *p*-chlorophenol and sulfur monochloride give a mixture of chlorophenol mono- and trisulfides¹¹⁷⁷ supports this assumption. In equation (4) the equilibrium will be displaced to the right if the sulfur is removed. As stated before, when the reaction is carried out at 60° or above, the sulfur, which is only slightly soluble in mustard gas, may separate out. In such cases the yield is high, 90% or better.^{546, 912} In the usual manufacture, at about 30°, much of the sulfur enters into combination. The production of sulfur dichloride according to equation (5) becomes more important. In actual practice, ethylene is passed into the reactor which is one third full of crude mustard gas from a previous run, of the composition given in Table 1.5, while sulfur monochloride is run in at the proper rate. This means that the sulfur chlorides, the sulfur, and the chloroethylsulfenyl chloride may react with each and every compound that is present. The concentration of the sulfenyl chloride is kept low by an excess of ethylene. Much of the sulfur which is liberated according to reaction (4) is thus used up.

Chloroethyl disulfide and trisulfide, $(\text{ClCH}_2\text{CH}_2)_2\text{S}_2$ and $(\text{ClCH}_2\text{CH}_2)_2\text{S}_3$, have been synthesized. As these are found in the crude, their reactions with sulfur are of interest. The trisulfide has been converted to the pentasulfide by heating with sulfur. Sulfur in excess of that required for the pentasulfide was deposited on standing. This sulfurization was accomplished far more readily with methyl tetrasulfide than with free sulfur, the reaction taking place at 35 to 40° instead of at 115 to 140°. The methyl tetrasulfide was reduced to the trisulfide which distilled out at a low pressure.⁵²⁴ This is of particular interest as it demon-

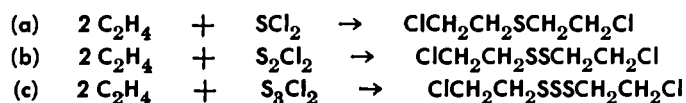
strates the transfer of sulfur from one polysulfide to another at a temperature near that used in the manufacture of mustard gas. At 90°, the pentasulfide took up more sulfur from methyl tetrasulfide. The composition of the product corresponded to the heptasulfide. On standing, it deposited some sulfur leaving an oil containing somewhat more sulfur than the hexasulfide.⁵²⁴

The disulfide could be sulfurized, but by no means as readily as the trisulfide, at least under these conditions. In view of this, it seems surprising that the amount of disulfide in the residue is as small as reported. The explanation seems to be the cleavage of the disulfide to the sulfenyl chloride by sulfur monochloride which was demonstrated experimentally.⁵¹⁷ In this reaction, sulfur monochloride appears to be acting as a chlorinating agent according to equation (3):

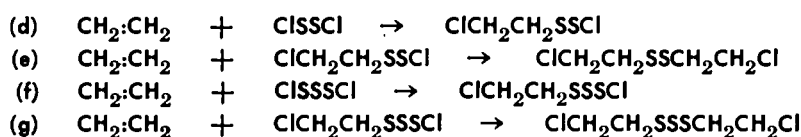


Toward the trisulfide, $(\text{ClCH}_2\text{CH}_2)_2\text{S}_3$, sulfur monochloride acts according to equation (4), sulfurizing instead of cleaving.⁵¹⁷

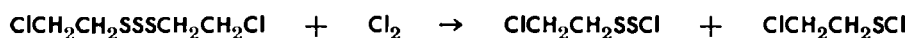
Over-all equations can be written for the formation of the di- and trisulfides, as well as for the monosulfide:



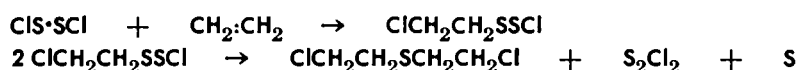
The steps in (a) have been clearly demonstrated. It is logical to assume that they are similar in (b) and (c):



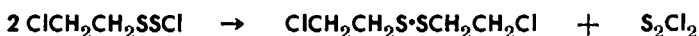
The postulated intermediate, $\text{ClCH}_2\text{CH}_2\text{SSCl}$, has been shown to be produced by the chlorinolysis of the trisulfide:



This reacts with an unsaturate to give a disulfide.⁵¹⁷ It seems safe to assume that the formation of the trisulfide involves the same steps. An early suggestion as to the formation of mustard gas involved the same intermediate:¹⁵²⁰



The formation of the disulfide rather than the monosulfide looks more reasonable:



Summing up, it can be said that enough reactions have been demonstrated to account satisfactorily for the compounds found in the Levinstein product. This is not saying that these are all, or even a majority, of the reactions that take place in the complex mixture in which there are so many reactive components. Any one of the compounds found may result from several different sequences of reactions. This applies particularly to the polysulfides which make up a large proportion of the residue. The polysulfides in the residue from the distillation of the monosulfide at above 100° may, or may not, be identical with those originally present in the crude.

Mustard gas, the chief product, is chlorinated by sulfur dichloride, by sulfur monochloride,^{911a} and by chloroethylsulfenyl chloride.⁵²¹ The product is 2-chloroethyl 1,2-dichloroethyl sulfide, $\text{ClCH}_2\text{CH}_2\text{SCHClCH}_2\text{Cl}$, whose decomposition into 2-chloroethyl 2-chlorovinyl sulfide, $\text{ClCH}_2\text{CH}_2\text{SCH}:\text{CHCl}$, and hydrogen chloride, is assumed to be the cause of the pressure that is built up in a closed container.¹³⁸⁷ The acid also causes corrosion.

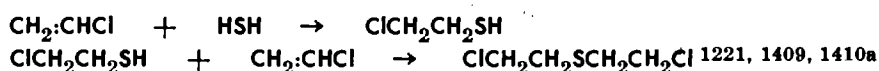
Mustard gas, being a monosulfide, does not combine with sulfur even on heating. The solubility of rhombic, or monoclinic, sulfur in it is 16.66% at 99° and only 1.48% at 24°. On cooling the sulfur separates out. Amorphous sulfur is far less soluble.¹⁴⁵⁸

To get pure mustard gas from the crude Levinstein product, it has been customary to distil in vacuum. When this is done batchwise, the material in the pot is kept hot for a long time and may be more or less altered. To avoid this, a continuous vacuum still has been devised.¹³³⁴

There seems to be some parallelism between the polysulfides in Levinstein mustard and the Thiokols. Thiokol A is a polymeric ethylene polysulfide, $(\text{CH}_2\text{CH}_2\text{S}_x)_n$, in which x usually has an average value between 3 and 4. Polymers can be made in which x is around 5 but sulfur blooms out of these on storage. These polymers can be stripped down to the disulfide by boiling them, in the latex form, with aqueous sodium hydroxide. In reverse, polymeric ethylene disulfide, $(\text{CH}_2\text{CH}_2\text{S}_2)_n$, which is a solid that can be powdered, is sulfurized by milling it with sulfur

and a vulcanization accelerator.³⁴⁵ At the other end of the scale, plastic sulfur, in which 10% of Thiokol A has been dissolved, remains plastic indefinitely.⁴⁰⁴

When a mixture of vinyl chloride and hydrogen sulfide, in liquid form, is irradiated with ultraviolet light at 25°, or below, addition takes place with the formation of a mixture of β-chloroethyl mercaptan and mustard gas:



Composition of Levinstein Mustard Gas

Representative analyses of a new and of an aged sample are given in Table 1.5.⁹⁰⁰

TABLE 1.5
Representative Analyses

	New	Aged
Acidity (as HCl), %	0.16	0.38
Iron (as FeCl ₃), %	0.22	0.41
Melting point	7.7°	7.6°
Density 20/4	1.351	1.340
Sulfur, %	33.46	30.70
Chlorine, %	36.35	38.42
Distillation analysis:		
Distillate, %	78.3	76.2
Residue, %	20.7	22.1
Loss, %	1.0	1.7
Melting point of distillate	10.2°	12.1°
Purity of distillate, %	88.5	93.8
Mustard content, % *	69.3	71.5

* Calculated from melting point assuming that the pure material melts at 14.4°.

These figures show the composition of the product as manufactured in steel containers on a large scale.⁹⁰⁰ It will be noted that in the new sample the sulfur and chlorine are present in practically equivalent amounts as they were in the sulfur monochloride. About 12% of the sulfur had separated out from the aged sample.

The distillation residue has been extensively investigated. All

of the evidence goes to show that it is a mixture of polysulfides and more or less free sulfur. The amount of the residue and its composition vary greatly according to the reaction conditions. If five out of six molecules of sulfur monochloride take up ethylene to form the monosulfide, $(\text{ClCH}_2\text{CH}_2)_2\text{S}$; five atoms of sulfur, two molecules of ethylene, and one of Cl_2S_2 will go into the residue which will have a composition represented by $(\text{ClCH}_2\text{CH}_2)_2\text{S}_7$. This is approximately the composition of the residue from the fresh sample in Table 1.5. In one study the residue had the composition of a pentasulfide and three fifths of its sulfur were oxidised to sulfuric acid.³¹⁰ In another, extraction of the crude by ethanol left a residue corresponding to $(\text{ClCH}_2\text{CH}_2)_2\text{S}_8$ from which ammonia precipitated half of the sulfur. Acetone dissolved out the trisulfide.¹⁰⁸⁷ In one case, Cellosolve took the pentasulfide from the residue, leaving a high-sulfur oil from which sulfur precipitated.⁵²⁴ The monosulfide can be hydrolyzed out of the crude by exhaustive washing with water, leaving a polysulfide residue.¹¹²⁰ Distillation of a sample of crude in a molecular still left a residue from which acetone extracted the tetrasulfide. This when passed through the same still gave some disulfide.⁹⁰⁰ In another study, boiling acetone extracted the trisulfide from the polysulfide residue, leaving sulfur.⁷⁷⁵ Of the constituents of the polysulfide residue the tri- and pentasulfides are reported oftenest and appear to be the most definite. The disulfide seems to be present in relatively small amounts, 2.23%¹⁰⁸⁷ and 4.3%.⁷⁷⁵ It may be that it is not formed in quantity or possibly part of it goes over with the monosulfide.

Physical Properties

Pure β,β' -dichloroethyl sulfide is a nearly colorless oily liquid but the crude is amber to brown. Many determinations have been made of its physical properties, a few of which are included in the general table of properties of chlorosulfides. This subject has been reviewed.⁶³⁴ The melting point has received much attention, since it is used in estimating the purity of a sample. There have been many determinations of its vapor pressure, particularly over the range of temperatures which might be encountered in summer or winter on the field of combat. In extremely cold weather it is scarcely volatile enough to be effective.

Recent determinations give 14.5° as the melting point.^{459, 525, 1413} Other values are 14.45° ,⁷⁷⁵ 14.4° ,^{945, 1011b, 1012, 1148} 13.82° ,¹⁴⁵⁸ 13.5° ,^{341, 1085} 12.85° ,⁶³⁴ 12° ,¹³²³ and 11° .¹³⁰⁹ It boils with slight decomposition under atmospheric pressure, b_{760} 217°C. , 422°F. ,^{634, 945, 956b} b_{727} 216.6° ,^{989c} $b.$ $215-7^{\circ}$,¹³⁰⁹ $b_{0.25}$ $54-7^{\circ}$,¹¹⁴⁸ b_1 $54-5^{\circ}$,⁵²² $75-80^{\circ}$,⁵²⁵ b_{10} 97° ,⁶³⁴ b_{14} 105° ,¹¹⁴⁸ b_{15} 105° ,⁶³⁴ 107° ,^{1011b, 1012} $108-9^{\circ}$,¹³²³ b_{18} 116° ,⁶³⁴ b_{20} $180-5^{\circ}$,¹³⁰⁹ b_{21} $98-9^{\circ}$,¹⁴⁵⁸ b_{24} 120° ,³⁴¹ b_{26} 117° ,⁶³⁴ b_{37} 122.5° .³⁴¹ There are numerous data on density: d $15/4$ 1.2790 ,¹⁴⁵⁹ d_{15} 1.275 ,¹³⁰⁹ 1.28 ,⁹⁴⁵ d $20/4$ 1.2741 ,¹⁴⁵⁹ 1.2746 ,^{1011b} d $21/4$ 1.2732 ,^{989c} d $23/4$ 1.2726 ,^{989c} d $25/4$ 1.2686 , d $90/4$ 1.1996 .¹⁴⁵⁹ The change in density for 1° is 0.001058 .¹⁴⁵⁹ There are data for the refractive index: n $15/\text{D}$ 1.53125 , n $15/\text{C}$ 1.52776 ,⁵⁴⁶ n $20/\text{D}$ 1.5281 ,⁵²² 1.5270 ,^{1011b} 1.5262 .⁵²⁵

The surface tension ^{989c} against its own vapor is 42.82 dynes, against water 28.36 , against $0.1N$ hydrochloric acid 28.90 and against sodium hydroxide 12.78 .⁶⁰⁷ The addition of 1% Alkaterge-0 lowers the surface tension of mustard gas and the interfacial tension between it and water.¹²¹⁵ The latent heat of fusion is 25 cal./g.; m.p. lowering 6.5° (1 mole solute per 1000 g. solvent).⁵¹ The compressibility at 0, 1000, and 2000 megabars is respectively 0.0000495 , 0.0000344 , and 0.0000239 . The melting point is raised from 13.9° at 1 megabar to 38.9° by increasing the pressure to 1800 megabars. The dipole moment is 1.76×10^{-18} .^{984c, 989a} The ultraviolet light absorption has been studied.^{704, 984a, 984b, 988, 989b}

Since the vapor pressure at a given temperature determines the maximum concentration that can be present in the air, it is a critically important property. The vapor pressure of mustard gas at 10° is 0.05 mm.,⁹⁴⁵ 0.055 mm.,¹⁰¹² while at 40° it is 0.45 mm.^{945, 1012} Determinations have been made from 0° to 60° .¹⁰¹² The value at any temperature is given by the formula:

$$\log_{10} p = 8.3937 - \frac{2734.5}{T}$$

From the vapor pressure the "volatility," which is the weight in milligrams per liter present in the air, is calculated by the formula:

$$V = \frac{M \times 273 \times p \times 1000}{22.4(273 + t)760} \quad 1226$$

where V is the volatility, p is the vapor pressure in millimeters of mercury at the temperature t on the centigrade scale and M

is the mole weight, which for mustard gas is 159.03 g. The observed and calculated values of the vapor tensions and the volatilities calculated from the calculated vapor pressures are given in Table 2.5.

TABLE 2.5

Temperature °C.	°F.	Vapor Pressure		Volatility milligrams/liter
		Observed	Calculated	
0	32	0.035	0.024	0.224
10	50	0.055	0.054	0.49
15	59	0.075	0.079	0.70
20	68	0.115	0.115	1.00
25	77	—	0.165	1.41
30	86	0.225	0.23	1.94
35	95	—	0.33	2.71
40	104	0.45	0.45	3.67
50	122	0.83	0.85	6.71
60	140	1.55	1.52	11.64

These values of the vapor pressure are higher than those formerly given.^{634, 822} Although the volatility of mustard gas is low, its toxicity is so great that air saturated with it even at 0°C. would cause death if breathed for a few minutes.

The vapor pressure has been determined by comparison with a standard liquid, diphenyl ether.¹¹⁸ Later determinations, which give considerably lower values, conform to the equations:

$$\begin{aligned}\log_{10} p &= 38.525 - 4500/T - 9.26 \log_{10} T^{77} \\ \log_{10} p &= 9.4819 - 3117.2/T^{117}\end{aligned}$$

At 30° the observed pressure was 0.1575 mm. instead of 0.225 mm. in Table 2.5.

Measurements by the static method between 70 and 120° are reproduced by the equation:⁶⁶⁹

$$\log p = 8.181 - 2625/T$$

Another equation is:¹¹⁴⁸

$$\log p = 9.31768 - 3062.5/T$$

The latent heat of vaporization is 14,420 cal. at 14.4° and 14,300 at 54°. The latent heat of fusion is 4,200 cal. per mole.⁷⁷

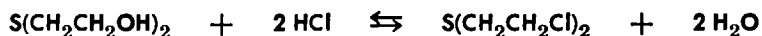
The rate of vaporization has been compared with those of several other liquids.^{984d}

The solubility of mustard gas in water and in 1 *M* sodium chloride solution are 0.92 and 0.5 g. per liter. Certain detergents increase the solubility.^{638a} The partition coefficient between xylene and water is approximately 200.⁸⁸¹ These values must be taken as approximations only, since hydrolysis is taking place while the measurements are being made. The critical solution temperature is 19° for ligroin, 20.4° for gasoline, 25.6° for kerosene,^{141, 1388} 38.6° for 92.5% alcohol, and 15.6° for absolute alcohol.¹³⁸⁶ The hydrocarbons and the alcohol can be used for extracting pure mustard gas from the crude Levinstein product. Extraction with low-boiling hydrocarbons can be made to give a material of 92.5% purity with 95% recovery.¹⁰¹³

Reactions of Mustard Gas

HYDROLYSIS

Mustard gas is readily hydrolyzed by water.^{1196, 1197} This is the reversal of the reaction by which it is made from thiodiglycol:



On account of the slight solubility of the dichlor-compound in water (0.48 g. per liter) and its high density, which causes it to settle out quickly, it may remain in contact with water for a long time with only slight decomposition. By agitating it with hot water, neutralizing the acid, and concentrating under diminished pressure, a practically quantitative yield of thiodiglycol can be obtained.¹⁷⁵ The hydrolysis is practically complete when the vapor is passed through water at 70°. ^{985b} The hydrolysis at 0°, 10°, 20°, 30°, and 37.5° has been found to be monomolecular. A method of analysis was developed depending on the hydrolysis.⁶⁷³ The reaction is monomolecular with respect to the mustard gas and independent of its concentration.¹⁰⁵³ The half life in water at 25° is 7 to 8 minutes. The hydrolysis reaches 96% in 40 minutes. The vapor is not hydrolyzed in moist air.¹¹²⁴

In a hydrolysis study the amount of mustard gas in solution was determined indirectly. After shaking, an aliquot of the aqueous layer was titrated immediately for hydrochloric acid and another after the hydrolysis was complete. The difference gave

the amount in solution. The hydrolysis is a two-stage reaction.^{1467b} The rate at 25° in alkaline solution is $dC/dt = 0.304(M)$ where (M) is its concentration. In hydrochloric acid this is the smaller the higher the concentration of the chloride ion. The solubility is given as 0.68 g. per liter at 25°. In aqueous alcohol, the rate is pseudo-unimolecular.^{986, 987} There have been more recent studies of the solubility and rate of hydrolysis. In one of these, use was made of the conductivity of the solution. The rate doubles for a rise of 5°. The solubility at 20° was found to be 0.75 g. per liter.¹³⁵⁶ The addition of hydrotropic substances, particularly egg lecithin,¹⁴⁹⁵ markedly increases hydrolysis and solubility. When the amount of water used is small, a complex mixture of sulfonium chlorides is formed along with the thiodiglycol. When mustard gas is shaken with a dilute solution of sodium silicate, each drop becomes surrounded by an envelop of silicic acid⁸⁰⁷ and thiodiglycol passes into solution.

The reactions of mustard gas in aqueous solution have been reviewed.¹⁰⁵²

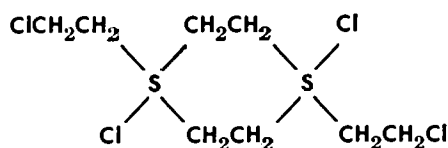
One of the mysteries about mustard gas is the way it escapes hydrolysis. Three cases of food poisoning from cod roe contaminated by mustard gas dumps in the Baltic have been reported. The roe gave a positive test by the auric chloride method.⁶⁵²

Early attempts to stop the hydrolysis at the half-way product, $ClCH_2CH_2SCH_2CH_2OH$, were not successful,^{73, 340} but it has been accomplished.¹⁰⁵³ This compound has been prepared by the reaction of thionyl chloride on an excess of thiodiglycol^{87, 583a, 1318} and by the addition of mercaptoethanol to vinyl chloride under the influence of ultraviolet light.^{525, 1217} A 72% yield has been obtained by letting a cold methanol solution of sodium hydroxyethylmercaptide, $HOCH_2CH_2SNa$, and a large excess of ethylene chloride stand for some days.¹²⁶⁸

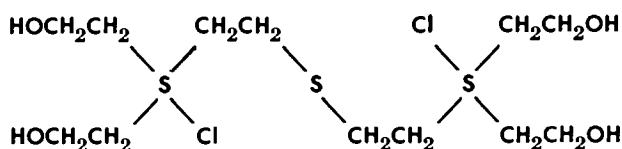
Semimustard, $ClCH_2CH_2SCH_2CH_2OH$, is an unstable liquid.^{525, 1217} Except when in solution, it exists as an equilibrium mixture of the monomer and the sulfonium dimer.^{583a} Its toxicity has been compared to that of mustard gas.³² It gave temporary relief to ten out of thirty-one cancer patients.¹²⁶⁸

The hydrolysis of mustard gas is complicated, involving a series of sulfonium salts. It can form sulfonium salts with

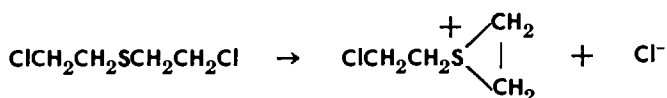
itself in at least two ways. One of these,



is postulated as an intermediate in the formation of dithiane, but is not involved in hydrolysis. Mustard gas does combine readily with thiodiglycol. When the two are shaken together with a limited amount of water at room temperature, a crystalline sulfonium salt is formed: ^{638b, 1398}



Kinetically the hydrolysis has been explained by considering that the rate determining reaction is the assumption of the sulfonium chloride structure:

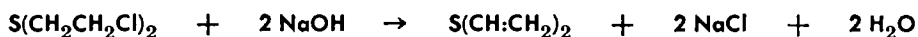


The three-membered ring is rapidly hydrolyzed to semimustard and then to thiodiglycol.⁸⁷ As soon as these are present, the formation of sulfonium salts goes on progressively. Several of these salts have been isolated and identified as their picryl sulfonates. The structures of others have been deduced from their reaction products.^{1312, 1321}

The hydrolysis of semimustard is about 50% more rapid than that of mustard and is believed to proceed similarly.^{583a, 1053}

Judged by the reaction with radioactive bromine ions, the chlorine atoms of β,β' -dichloroethyl sulfide are about twenty times as reactive as those of dichloroethyl ether.²³⁹

Treating mustard gas with strong alkali, particularly in alcohol solution, leads to the removal of hydrogen chloride as well as to the substitution of ethoxyl for chlorine: ⁶²⁶



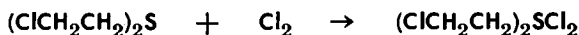
The yield may be increased by direct distillation.⁷³ It is better to prepare the vinyl sulfide from β,β' -dibromoethyl sulfide.¹²²⁰ A mixture of the following products may be obtained: vinyl

sulfide, $S(CH:CH_2)_2$, β -chloroethyl vinyl sulfide, $ClCH_2CH_2SCH:CH_2$, β -ethoxy vinyl sulfide, $EtOCH_2CH_2SCH:CH_2$, $EtOCH_2CH_2SCH_2CH_2OH$, and β,β' -diethoxyethyl sulfide, $S(CH_2CH_2OEt)_2$.³⁴⁰

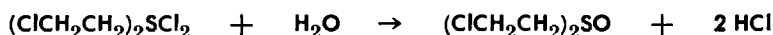
CHLORINATION

The chlorination of mustard gas has been studied extensively and numerous products have been isolated.^{351, 830, 911a, 1011a, 1097} The course the reaction takes depends on conditions. There are many possibilities, most of which seem to have been realized.

At 0° in carbon tetrachloride solution, mustard gas takes up chlorine:



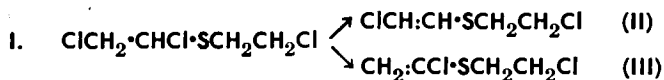
This separates as white needles. In aqueous alcohol it is hydrolyzed to the sulfoxide:



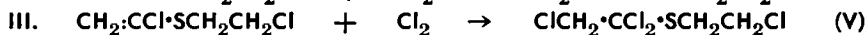
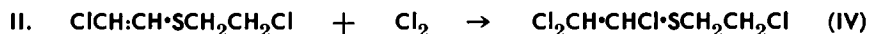
It is unstable; at room temperature, the chlorine migrates to the alpha carbon:⁸³⁰



This α,β,β' -trichloroethyl sulfide loses hydrogen chloride on heating, or even on standing at room temperature:

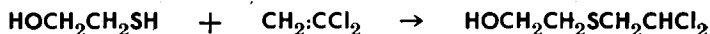


Both of these take up chlorine:



According to another investigator, the loss of hydrogen chloride from I leads to two forms of II, $II\alpha$ and $II\beta$, probably *cis* and *trans*.⁹⁸³

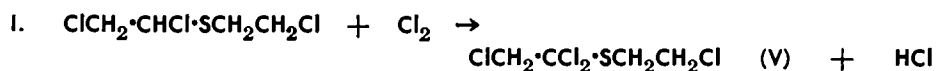
An isomer of I has been prepared by adding mercaptoethanol to vinylidene chloride and treating the product with thionyl chloride:



This was hydrolyzed to 2-hydroxyethylmercaptoacetaldehyde which was shown to be identical with a synthetic product.⁵²¹

Curiously enough II α and II β give this same aldehyde on hydrolysis.⁹⁸³

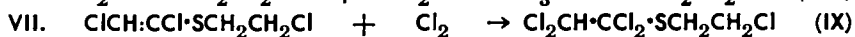
Chlorination of I may also give V:



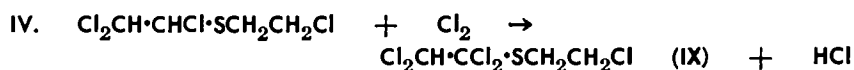
In vacuum distillation:



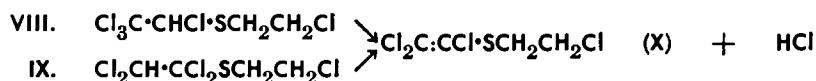
By addition of chlorine:



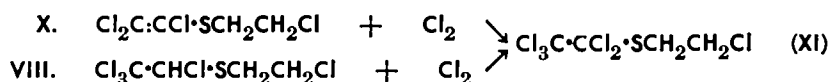
By chlorination:



In vacuum distillation:



By adding chlorine to X or chlorinating VIII.

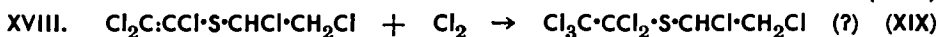
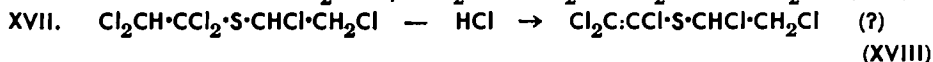
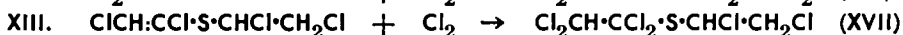
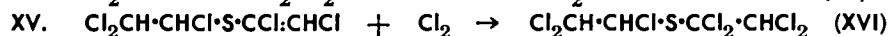
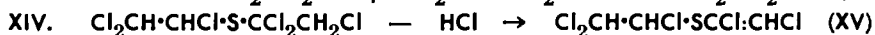
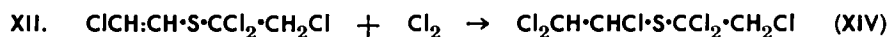


Further chlorination leads to cleavage of the molecule ending up with hexachlorethane and sulfur chloride.

Under different conditions, the other chlorethyl group may be attacked. Two compounds have been isolated,¹⁰⁹⁷



By addition of chlorine, subtraction of hydrogen chloride and further addition of chlorine:



Chlorination of mustard gas with Cl_2NCOOMe gives the unstable $\alpha,\alpha',\beta,\beta'$ -tetrachloro-derivative.^{173, 270} The product from sulfur monochloride and trichloroethylene is perchlorovinyl sulfide, $(\text{CCl}_2:\text{CCl})_2\text{S}$.^{408, 661}

The physical properties of the more stable compounds are given in Chapter 7. Other compounds have been isolated but their structure has not been determined: $\text{C}_2\text{H}_2\text{Cl}_3\text{SCl}$, $b_{3.5}$ 53.8° , $\text{C}_4\text{H}_3\text{Cl}_7\text{S}$, $b_{5.5}$ $144-6^\circ$; d 20/4 1.7373, d 25/4 1.7325; n 20/D 1.5739 and $\text{C}_4\text{H}_3\text{Cl}_5\text{S}$, b_{15} $134-4.5^\circ$; d 20/4 1.6248, d 25/4 1.6195.¹⁰⁹⁷

By the addition of chlorine to vinyl sulfide, we obtain $\text{CH}_2:\text{CHS}\cdot\text{CHCl}\cdot\text{CH}_2\text{Cl}$ and $\text{ClCH}_2\cdot\text{CHCl}\cdot\text{S}\cdot\text{CHCl}\cdot\text{CH}_2\text{Cl}$.¹⁷ On distillation under atmospheric pressure, the tetrachloro-compound loses hydrogen chloride, leaving β,β' -dichlorovinyl sulfide, $(\text{ClCH}:\text{CH})_2\text{S}$.^{334, 787}

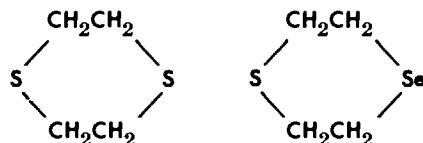
Vinyl sulfide is oxidised by perbenzoic acid to the sulfoxide, $(\text{CH}_2:\text{CH})_2\text{SO}$, b_{18} 81° ; d 17/4 1.0867, d 20/4 1.084; n 20/D 1.5100 and the sulfone, $(\text{CH}_2:\text{CH})_2\text{SO}_2$, b_8 102° ; d 20/4 1.1794; n 20/D 1.4799.^{850b}

The chlorovinyl chloroethyl sulfides II and III have been oxidised: $\text{ClCH}:\text{CH}\cdot\text{SO}_2\cdot\text{CH}_2\text{CH}_2\text{Cl}$, m . $109-11^\circ$, $\text{CH}_2:\text{CCl}\cdot\text{SO}\cdot\text{CH}_2\cdot\text{CH}_2\text{Cl}$, b_8 104° , $\text{CH}_2:\text{CCl}\cdot\text{SO}_2\cdot\text{CH}_2\text{CH}_2\text{Cl}$, d 20/4 1.4366; n 20/D 1.5238.⁸⁵¹

OTHER REACTIONS

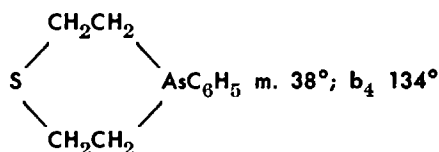
With bromine, mustard gas gives an unstable addition compound, $(\text{ClCH}_2\text{CH}_2)_2\text{S}\cdot 2\text{Br}_2$. This gives off bromine, leaving $(\text{ClCH}_2\text{CH}_2)_2\text{S}\cdot\text{Br}_2$, m . 44° .⁵⁴⁶

With sodium sulfide and selenide, poor yields of the cyclic compounds are obtained: ^{545, 577}

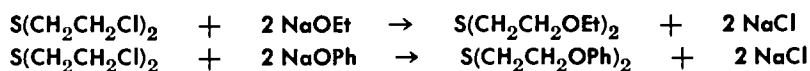


Sodium selenomercaptide gives $\text{S}(\text{CH}_2\text{CH}_2\text{SeEt})_2$.¹²⁷³ A polymer is formed from mustard gas and the sodium salt of bentonite clay.³⁷¹ With sodium, or calcium, disulfide a polymeric resin is formed.⁷⁴⁰ With sodium tetrasulfide, the product is $(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{S}_4)_n$, a polymer of the Thiokol type.^{2, 1081}

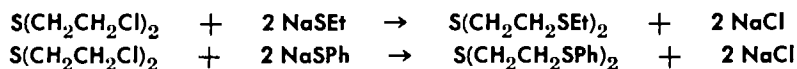
With magnesium phenylarsine a cyclic compound is obtained:⁷³³



With sodium alcoholates and phenates, ethers are formed, but the yields are only fair:⁶²⁶

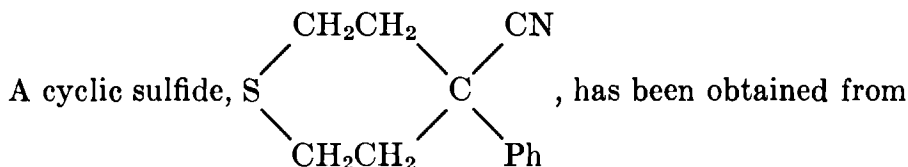


With the corresponding sulfur compounds, almost quantitative yields are obtained:⁶²⁶



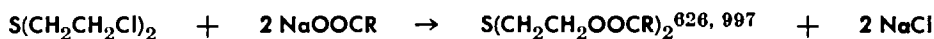
The formation of the dimethyl derivative, $\text{S}(\text{CH}_2\text{CH}_2\text{SMe})_2$, which is harmless and can be distilled readily, has been used to characterize mustard.⁹⁴¹ In the presence of zinc dust, mustard gas reacts with alcohols to form ethers, $\text{S}(\text{CH}_2\text{CH}_2\text{OR})_2$. These are probably from the addition of the alcohols to vinyl sulfide.^{808a}

By way of malonic or acetoacetic ester, the acid $\text{S}(\text{CH}_2\text{CH}_2\text{CH}_2\text{COOH})_2$ may be synthesized.³⁴¹



mustard gas and phenylacetonitrile with the aid of sodamide.⁴¹⁹ This is the sort of compound which Victor Meyer wanted to make with malonic ester.^{956a}

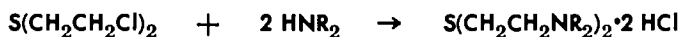
With salts of organic acids, esters of thiodiglycol are produced:



The reaction products with sodium alginates and pectinates may be of this class.³⁷² With an excess of sodium N-diethyldithiocarbamate, the half ester, $\text{Et}_2\text{NCSSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl}$, is formed.^{1089a}

Mustard gas does not act on aluminum, lead, brass, iron, bronze, zinc or tin at room temperature, but at 100° attacks all of these except aluminum, lead, and brass.⁵⁴⁶ This is true of the pure substance. In steel containers, it decomposes at 150°F.⁹⁰⁰ To determine the stability of the crude material, a 300 cc. sample is stored in a 75 mm. steel shell closed by a rubber stopper carrying a manometer. This is kept at 60° and the pressure read from time to time.^{460, 1387} One sample of crude mustard gas, stored in a steel container at 65°, deposited 30% of solids within 21 days. With 1% of hexamethylene tetramine, the solids amounted to only 3% in 110 days.⁸⁸³ According to another report, this material prevents gas evolution but does not inhibit decomposition.⁵¹⁴

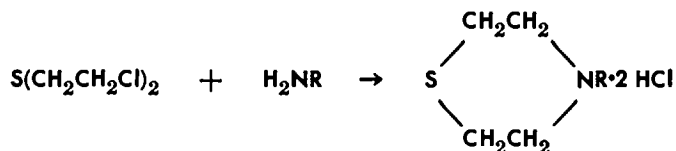
With a secondary amine, mustard gas reacts simply: ⁸³¹



One amine group may be lost:



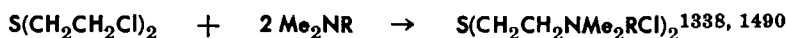
With ammonia or a primary amine a cyclic thiomorpholine results: ^{226, 292a, 626, 831}



1,4-Thiazane, $\text{S}(\text{CH}_2\text{CH}_2)_2\text{NH}$ boils at 169°.³⁴¹ Cetyl amine, mustard gas, and sodium carbonate refluxed in ethanol give N-cetylthiomorpholine, m. 78°. The N-dodecyl and N-tetradecyl compounds are obtained similarly.^{611, 1039} Ethyl aminoacetate reacts similarly in the presence of sodium acetate.⁴⁷³ N-alkylthiomorpholines are claimed as oxidation inhibitors.⁹⁹⁸ Two molecules of the amine may react with one of the mustard: ²⁸³



Mustard gas unites with tertiary amines to form quarternary ammonium salts:

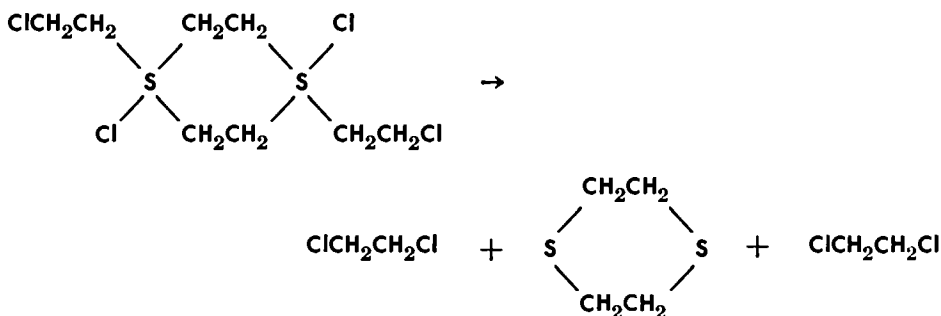


Mustard gas forms a complex with thiourea.¹¹⁹² When it is heated with a concentrated aqueous solution of thiourea, the

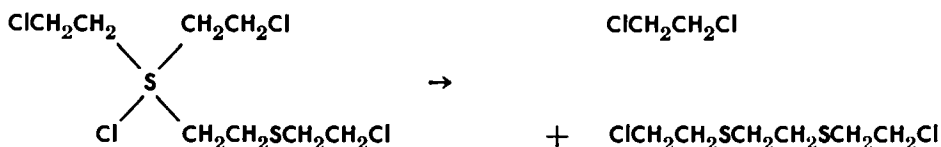
formation of the isothiuronium salt is complete in about 15 minutes. This is decomposed by aqueous sodium hydroxide. Acidification liberates the mercaptan, $S(CH_2CH_2SH)_2$, which is obtained in 84% yield.¹¹⁵²

SULFONIUM

The fact that sulfonium compounds are involved in the hydrolysis of mustard gas has been mentioned before. It is assumed that it forms sulfonium compounds with alkyl halides, but these are unstable; the products that can be isolated are derived from dithiane.^{1034a} At a high temperature, it appears to form a sulfonium complex with itself which then decomposes:^{101, 105}



The formation of sesqui-mustard and a still higher $S(CH_2CH_2SCH_2CH_2Cl)_2$ can be formulated similarly:⁵²⁰



When mustard gas is treated with potassium cyanide, the product is $(\cdot\text{CH}_2\text{SCH}_2\text{CH}_2\text{CN})_2$, which is derived from sesqui-mustard.^{341, 342}

The sulfonium chloride, $(\text{HOCH}_2\text{CH}_2)_3\text{S}\cdot\text{Cl}$, from ethylene chlorhydrin and thiodiglycol can be converted to the trichloro-derivative, $(\text{ClCH}_2\text{CH}_2)_3\text{SCl}$. This is not vesicant. Its water solution is neutral but soon becomes acid.^{430, 1116}

The thermal decomposition of mustard gas has been studied at 180, 220, 350, and 450°. Ethylene is evolved at all temperatures, ethylene dichloride and dithiane appear at the two lower temperatures and vinyl chloride and hydrogen sulfide at the two

higher temperatures. The amount of hydrogen chloride increases as the temperature is raised.¹⁴⁶¹

A quadrivalent sulfur derivative is formed with chloramine-T,^{911b} $(\text{ClCH}_2\text{CH}_2)_2\text{S}:\text{NSO}_2\text{C}_6\text{H}_4\text{CH}_3$, m. 144.5° . This is mildly toxic to rats.⁷⁴⁹

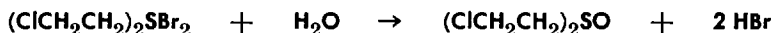
With iodine trichloride, there is an addition product which has been written:¹⁶⁰



It forms complexes with salts of platinum, gold, and other heavy metals. These are discussed under Detection. The complex, $(\text{ClCH}_2\text{CH}_2)_2\text{S}\cdot\text{PdCl}_2$, is mildly toxic to rats.⁷⁴⁹

SULFOXIDE

When mustard gas is dropped into concentrated nitric acid, there is an energetic reaction with the formation of a light-green solution. The basic salt $(\text{ClCH}_2\text{CH}_2)_2\text{SO}\cdot\text{HNO}_3$, or $(\text{ClCH}_2\text{CH}_2)_2\text{S}(\text{OH})\text{NO}_3$, seems to be present. On dilution with water, the sulfoxide, $(\text{ClCH}_2\text{CH}_2)_2\text{SO}$, m. 109.5° , separates out.⁶²⁸ Neutralization of the acid mother liquor causes more of it to come down. The basic nitrate can be obtained in crystalline form. It is unstable on the water bath. Continued heating with concentrated nitric acid may take it all the way to the sulfonic acid, $\text{ClCH}_2\text{CH}_2\text{SO}_3\text{H}$.⁴⁸⁶ The bromine addition compound, $\text{Br}_2\text{S}(\text{CH}_2\text{CH}_2\text{Cl})_2$, is readily hydrolyzed to the sulfoxide:⁵⁴⁶



Hydrogen peroxide, in acetic acid at room temperature, is effective.^{1309, 1323} Oxidation by perbenzoic acid is quantitative.^{850a, 850b} If mustard gas, dissolved in an unsaturated oil, such as poppyseed oil, is exposed to the air for 2 weeks, the sulfoxide is formed.^{174, 1185} If it is added to turpentine that has been exposed to the air, the sulfoxide crystallizes out.¹⁴²⁷ It can be assumed that in these cases, the oxidation was by hydroperoxides. When mustard gas is mixed with a slight excess of cumene hydroperoxide and let stand for several days, the sulfoxide crystallizes out.¹¹⁵⁸

The sulfoxide crystallizes well from water, 100 g. of which dissolves 1.2 g. at 20° and an unlimited amount at 100°. The solubility in 95% alcohol is 4.3 g. per 100 cc. It is much less stable than mustard gas or its sulfone. A sample distilled at 18 mm. was largely decomposed; the distillate contained mustard gas and sulfoxide, but no sulfone.⁶²⁶ A sample kept in a glass-stoppered bottle decomposed on storage for some months.¹¹⁵⁸

Hydrolysis in neutral solution is extremely slow; a boiling aqueous solution gives no precipitate with silver nitrate. It is hydrolyzed by alkali to the dihydroxysulfoxide, $\text{OS}(\text{CH}_2\text{CH}_2\text{OH})_2$, b_{20} 295–310°, and the cyclic 1,4-thioxane sulfoxide, $\text{OS}(\text{CH}_2\text{CH}_2)_2\text{O}$, m. 25°, b_{15} 147°.²⁶¹

In general, the sulfoxide does not react as regularly, or give such good yields of derivatives, as the sulfide or the sulfone, but with mercaptans or thiophenols in alkaline solution, fair yields are obtained. $(\text{BuSCH}_2\text{CH}_2)_2\text{SO}$, m. 25°, $(\text{PhSCH}_2\text{CH}_2)_2\text{SO}$, m. 121°, and $\text{PhN}(\text{CH}_2\text{CH}_2)_2\text{SO}$, m. 123.5°, have been recorded.⁶²⁶ It is said to react with sulfhydryl groups in the skin.⁵⁷³

Hypochlorous acid oxidises and chlorinates mustard gas to the tetrachlorosulfoxide, $(\text{ClCH}_2\text{CHCl})_2\text{SO}$, m. 121°. The same compound is a by-product from the washing of crude acetylene with chlorine water. This, heated with sodium bicarbonate, loses hydrogen chloride, giving β,β' -dichlorovinyl sulfoxide, $(\text{ClCH}:\text{CH})_2\text{SO}$, b_{14} 99–110°, which can be reduced by zinc and dilute acid to the sulfide, $(\text{ClCH}:\text{CH})_2\text{S}$. Oxidation of the tetrachloro compound may give the unsaturated sulfone, $(\text{ClCH}:\text{CH})_2\text{SO}_2$, $b_{0.35}$ 60–63°, or the sulfonic acid, $\text{ClCH}_2\cdot\text{CHClSO}_3\text{H}$.¹⁰⁰⁷ The rates of oxidation by hypochlorite at several values of pH have been measured.^{668b}

By adding halogens to vinyl sulfoxide, the tetrachlorosulfoxide, m. 122.5°, and the tetrabromosulfoxide, $(\text{BrCH}_2\cdot\text{CHBr})_2\text{SO}$, m. 120.8°, have been obtained and from them the vinyl derivatives, $(\text{ClCH}:\text{CH})_2\text{SO}$, m. 15°, b_{10} 87°, d_{20} 1.417, and $(\text{BrCH}:\text{CH})_2\text{SO}$, m. 42°.⁸⁰⁹

The toxicity of the sulfoxide is low.^{578, 818c} It has no apparent effect on a man's skin.⁵⁷⁸ As shown by the use of radioactive sulfur, it is distributed widely and rapidly in the animal body, but the highest concentrations are found in the kidneys and lungs.^{182c} When the toxicity is determined by injection in mice,

the story is quite different. Then the sulfoxide is practically equivalent to the sulfide.⁹²¹

SULFONE

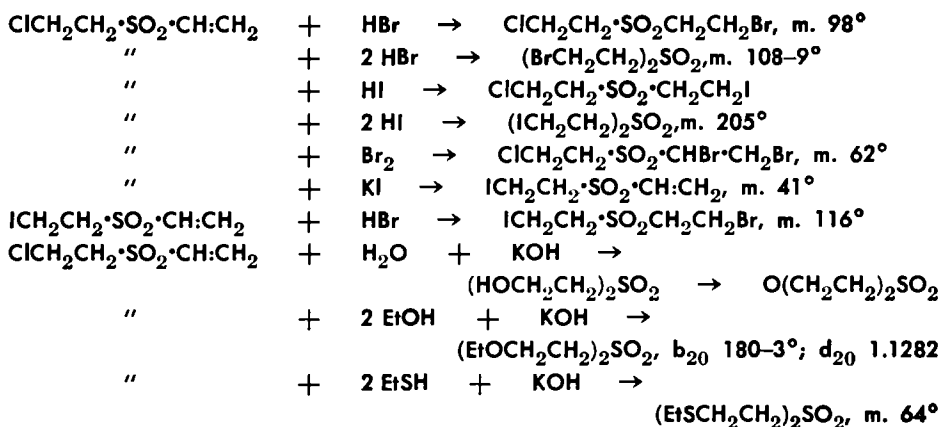
β,β' -Dichloroethyl Sulfone

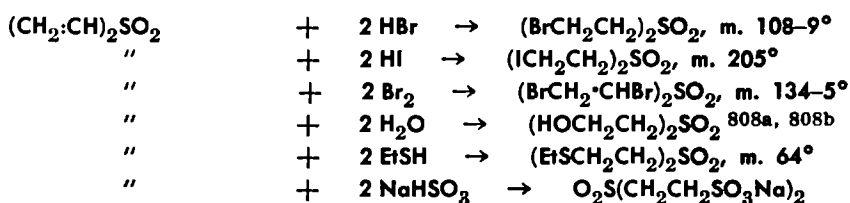
Strong oxidation carries mustard gas, or the sulfoxide, to the sulfone,^{626, 850a, 850b, 1309, 1323} $\text{O}_2\text{S}(\text{CH}_2\text{CH}_2\text{Cl})_2$, m. 56.0° , b_{20} 183° , b_{760} about 230° with decomposition. In 100 cc. of water 0.6 g. dissolves at 20° and 2.4 g. at 100° . At 20° , 100 cc. of alcohol dissolves 7.1 g.⁶²⁶ Ozone oxidises mustard gas, in chloroform solution, to the sulfone.^{151a}

In boiling water, it gives a slight precipitate with silver nitrate.⁶²⁶ In aqueous alkali, it is hydrolyzed to a mixture of the dihydroxy sulfone, $\text{O}_2\text{S}(\text{CH}_2\text{CH}_2\text{OH})_2$, m. $57\text{--}58^\circ$,^{626, 850b} and the cyclic 1,4-thioxane sulfone, $\text{O}_2\text{S}(\text{CH}_2\text{CH}_2)_2\text{O}$.²⁶¹ In methanol and ethanol with alkali the corresponding alkoxy derivatives are formed, $\text{O}_2\text{S}(\text{CH}_2\text{CH}_2\text{OCH}_3)_2$, b_{25} 195° , and $\text{O}_2\text{S}(\text{CH}_2\text{CH}_2\text{OC}_2\text{H}_5)_2$, b_{15} 144° .²⁶¹

The sulfone is hydrolyzed about ten times as rapidly as a normal alkyl halide, but far from as rapidly as mustard gas.¹⁵⁶ Refluxed with aqueous sodium carbonate, it goes to the cyclic thioxane sulfone.^{479b} Mixed with a tertiary amine,¹³¹¹ or boiled with water and calcium carbonate,⁴⁸⁰ the sulfone is converted into divinyl sulfone. With zinc dust or with zinc oxide in alcohol, the sulfone loses hydrogen chloride in two stages giving $\text{ClCH}_2\text{CH}_2\cdot\text{SO}_2\cdot\text{CH}:\text{CH}_2$, b_{22} $155\text{--}156^\circ$, and $(\text{H}_2\text{C}:\text{CH})_2\text{SO}_2$, b_8 102° , b_{20} $118\text{--}121^\circ$; $d_{20/4}$ 1.1794; $n_{20/D}$ 1.4799.^{808b}

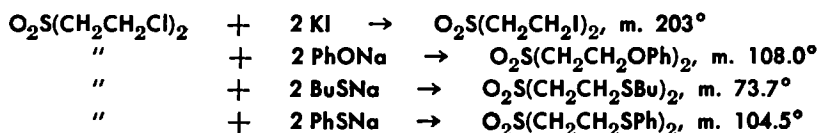
Both of these are reactive: ^{808b}



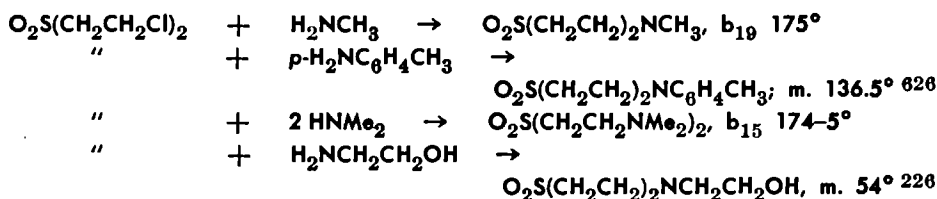


Oxidation of $(\text{EtSCH}_2\text{CH}_2)_2\text{SO}_2$ gives $(\text{EtSOCH}_2\text{CH}_2)_2\text{SO}_2$, m. 139°, and $(\text{EtSO}_2\text{CH}_2\text{CH}_2)\text{SO}_2$, m. 222°.

The sulfone of mustard gas reacts with sodium and potassium salts as does the parent compound: ⁶²⁶



It reacts also with primary and secondary amines: ⁸³¹



Physiological Effects

The sulfone is more toxic than the sulfoxide,^{651, 1371b} but less so than mustard gas itself. It produces edema, but not loss of weight or death.^{1371b} On the skin, it produces blisters and persistent sores, but on account of its low vapor pressure there is little danger in handling it.⁶²⁶ The percutaneous administration of the sulfone is said to be at least as toxic as mustard gas to a guinea pig.^{818c} By the use of radioactive sulfur, its distribution to all parts of the body was found to be rapid. The highest concentrations were found in the kidneys and lungs.^{182c} The bleeding time of a rabbit is lengthened if the wound is washed with a solution of the sulfone. This is counteracted by British antilewisite "B.A.L." ⁶⁸² The pyruvate oxidase system is attacked selectively ^{1089d} and urease is inactivated.⁴⁷² Other effects have been noted.^{58a, 58b} The reaction is slower with native than with denatured ovalbumin. This is attributed to the formation of the sulfhydryl groups.⁶³

Detection and Estimation of Mustard Gas

Naturally much attention has been given to the detection and estimation of mustard gas.^{31, 324, 366, 376, 405, 410, 461, 660, 671a, 671b, 773, 838, 843, 855, 1033, 1044, 1194, 1213, 1361, 1494} Passing air containing mustard gas through water causes a change in the pH, which can be detected by bromphenol blue. As most of the war gases are hydrolyzable, this is not specific.⁷⁸⁸ Contaminated air can be passed through a hot tube and the sulfur dioxide absorbed and determined.^{933, 1149} Any hydrogen sulfide or sulfuric acid present should be taken out before it is burned.⁴¹⁸ By the Beilstein flame method, chlorine, as well as the sulfur dioxide, can be detected.^{416, 1402} As little as 0.005 mg. can be detected by the reduction of selenious acid. By comparison with standards, 0.01 to 0.1 mg. may be estimated.¹⁴⁸⁹ A suspected droplet can be decomposed by an electrically heated filament and the evolved gas brought in contact with starch iodate or alcoholic *o*-toluidine on paper.¹²⁷² Iodine trichloride is also a sensitive reagent.^{398b} Mustard gas may be wiped off objects with absorbent cotton which is then extracted with ether. The residue from this can be subjected to various tests.⁹²² Samples of contaminated soil may be extracted with ether.^{1335, 1477}

Advantage is taken of the fact that the diiodoethyl sulfide, $(\text{ICH}_2\text{CH}_2)_2\text{S}$, which is readily formed from mustard gas and sodium iodide, is very slightly soluble.^{586, 631, 1186} A blue color is produced when mustard gas adsorbed in silica gel is treated with a starch solution and hydriodic acid.^{43a}

Auric chloride forms a brown addition compound, $\text{AuCl}_3 \cdot \text{S}(\text{CH}_2\text{CH}_2\text{Cl})_2$, with mustard gas. This is relied on to detect small amounts which may be adsorbed on silica gel.^{110, 146, 186, 374, 393, 395, 396, 398a, 631, 672, 858a, 1044, 1050, 1059, 1077, 1172a, 1173, 1254a, 1255, 1313, 1477} This can be used with frozen skin sections.¹²⁸⁸ A solution containing 1% of phosphotungstic acid and 0.1% of auric chloride is useful for testing for mustard gas in water, down to one part in 200,000.^{1172b, 1173} Heavy metal salts, such as sodium iodoplatinate, may be utilized.^{18, 186, 395, 610, 691, 801, 866a, 1044, 1179, 1255} Palladium chloride changes color with mustard gas.^{631, 1255} It has been used on silica gel.³⁹⁵ The reaction is said to be specific.¹⁰⁵⁰

Mustard gas cuts down the fluorescence of an alkaline alcoholic

solution of fluorescein. This has been studied quantitatively.⁵¹¹

The vapor may be taken out of the air by activated carbon.^{376, 415, 741} From air saturated with mustard gas vapor, 100 g. of cocoanut charcoal adsorbs 47 cc. as liquid 28 cc. of which may be removed by passing pure air through it.¹² The sorptive capacities of a number of activated charcoals have been measured.¹²⁷⁴ Contaminated air is drawn through a tube containing silica gel. The gel from the tube is spread out on a plate and reagents applied for spot tests.⁴⁶¹

The vapor mixed with air that has been passed over bleaching powder gives unstable white crystals, m. 50–60°. ^{397b, 1315} In droplets mustard gas may be identified by its violent reaction with bleaching powder.¹⁷⁰

It is possible to estimate mustard gas in the presence of other sulfides by the frontal method of chromatographic absorption.⁶⁸⁸ The detection in medicines ⁷⁰⁸ and in sewage ⁵⁷⁹ has been described.

Color tests have been much sought after. Paper impregnated with an ammoniacal solution of silver nitrate and isatin turns red.⁷²⁹ Sudan red on ground chalk and sea sand turns red with several toxic gases. Treatment with ferric chloride changes the red to green only with mustard gas.^{858b} A green color is produced with a cupric salt under certain conditions.¹¹¹⁰ Several dyes are recommended.²⁷⁸ Contaminated air is drawn through a buffered solution of 2,6-dichlorophenol-indophenol. The color is discharged by as little as 20 to 30 gamma.^{957a} A paint that changes color has been prepared.⁸⁹³ Absorption spectra may be used for mustard gas and other war gases.^{984b, 985a}

Mustard gas is not polarographically active, but can be made so by treatment with an ammoniacal solution of a cobalt salt.¹⁹⁸ It decolorizes an acid solution of potassium permanganate, 0.015 g. per liter.¹¹⁰³ It can be determined by using a known amount of dichloramine-T and titrating the residual chlorine.^{583c, 777b} It can be titrated in dilute aqueous solution with 0.001 *M* bromine solution,^{1046c} with potassium iodate,¹²⁶¹ or with sodium hypochlorite.⁸³⁹ By the change of color due to the reduction of platinic salt it may be estimated colorimetrically.¹²⁶¹ Sodium mercuric iodide gives precipitates which may be collected in a graduated centrifuge tube.²³⁰ Mustard gas reacts with Nessler's

reagent.³⁶¹ It combines with cuprous chloride as a double salt, $[(\text{ClCH}_2\text{CH}_2)_2\text{S}]_2\cdot\text{Cu}_2\text{Cl}_2$.^{395, 1255} It is added to a standard solution of cuprous chloride the excess of which is titrated after filtering off the precipitate.⁶⁶⁵ Cuprous iodide also is useful.⁴⁰⁰ Automatic recorders for field use have been devised.^{47, 792} Biological tests have been proposed.^{362, 1046a, 1400} The purity of a sample of mustard gas is estimated from its melting point.⁹⁰⁰

Analogs and Homologs

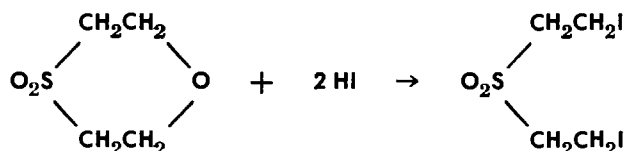
BROMIDE AND IODIDE

From thiodiglycol with phosphorus tribromide or better with hydrogen bromide, the bromine analog is obtained.^{226, 1323} The same reagents produce it from diethoxyethyl sulfide: ^{808a}



At 20°, its saturated vapor is about 0.4 mg. per liter. Its reactions are similar to those of mustard gas.¹¹⁹⁶ As it is a solid at room temperature and has a low vapor tension, it can be handled with little danger, and so it is a convenient substitute for mustard gas in making derivatives.²²⁶ Its physiological effects are milder.^{934, 1456} It is oxidised ^{226, 850b} to the sulfoxide, m. 100–101.4°, ^{850b} and the sulfone, m. 111–112°, ²²⁶ which may be prepared by the addition of hydrobromic acid to vinyl sulfone.^{808b}

The iodide, $\text{S}(\text{CH}_2\text{CH}_2\text{I})_2$, is prepared from mustard gas and sodium iodide in alcohol or in acetic acid solution ^{586, 626} or by the addition of hydriodic acid to vinyl sulfide.¹⁷ Its reactions are similar to those of mustard gas. It can be oxidised to sulfoxide and sulfone. The sulfone has been obtained from thioxane sulfone and hydriodic acid: ⁵⁰⁸



Treating mustard gas with dry silver fluoride converts it to the fluorine compound, $(\text{FCH}_2\text{CH}_2)_2\text{S}$. This is oxidised by nitric acid to the sulfoxide, $(\text{FCH}_2\text{CH}_2)_2\text{SO}$, and by chromate to the sulfone, $(\text{FCH}_2\text{CH}_2)_2\text{SO}_2$.

TABLE 3.5

Properties of Dihalosulfides and Their Oxidation Products

	Sulfide	Sulfoxide	Sulfone
Difluoro-	— b ₃₆ 95–6°	m. 103°	m. 42° ⁹⁰⁶
Dichloro-	m. 14.4°; b. 217°	m. 109.5°	m. 56° ⁶²⁶
Dibromo-	m. 34°; ²²⁶ b ₁ 115.5° ¹¹⁹⁶	m. 101° ^{850b}	m. 112° ²²⁶
Diiodo-	m. 70° ^{808a}	m. 104.5° ⁶²⁶	m. 203° ⁵⁰⁸

All three of the sulfoxides melt within the range 101 to 110°, while the melting points of the sulfones go up rapidly. The density of the dibromosulfide is 2.05.¹¹⁹⁶

DI- AND TRISULFIDE

To help clear up the chemistry of the Levinstein process the disulfide, ClCH₂CH₂S·SCH₂CH₂Cl, has been prepared, starting with chlorhydrin and sodium disulfide. The glycol,^{104a, 350a, 524} (HOCH₂CH₂S·)₂, heated with concentrated hydrochloric acid gave the chloride, (ClCH₂CH₂S·)₂, b₃₀ 155°; d 20/4 1.3375.^{104a} It has been made also by treating chloroethyl mercaptan, ClCH₂CH₂SH, with iodine.³⁵⁸ It has been isolated from the Levinstein product⁵²⁴ and has been synthesized by the addition of the chloride, ClCH₂CH₂SSCl, to ethylene.⁵¹⁷ Its vesicant action is about one third that of the monosulfide. With sodium phenate it gives the diphenoxy-derivative, PhOCH₂CH₂S·SCH₂CH₂OPh, m. 96–97°; ^{104a} compare PhOCH₂CH₂SCH₂CH₂OPh, m. 54.2°.

The trisulfide, (ClCH₂CH₂)₂S₃, is a white solid, melting at 31.5°, which can be distilled at 0.01 mm.⁵²⁴ The crystal structures have been determined for this^{348, 384} and for the iodotrisulfide, (ICH₂CH₂)₂S₃.^{348, 349}

α,α'-DICHLOROETHYL SULFIDE

α,α'-Dichloroethyl sulfide results when paraldehyde is treated with thionyl chloride¹⁰⁰⁸ or when hydrogen chloride is added to vinyl sulfide:



At 15 mm., it boils at 58.5° while β,β' -dichloroethyl sulfide boils at 107° .⁷³ The densities at 15° are α,α' 1.1972 and β,β' 1.2790. Its physiological action is only slight. It loses hydrogen chloride readily. It is oxidised by perbenzoic acid to the sulfoxide, $(\text{CH}_3\text{CHCl})_2\text{SO}$, b_{1-2} $68-70^\circ$; d 16/4 1.3142, d 20/4 1.3106; n 20/D 1.5089 and to the sulfone, $(\text{CH}_3\text{CHCl})_2\text{SO}_2$, $m.$ $78-80^\circ$.^{850b} By various reagents the α,α' -dichloroethyl sulfide can be converted into γ -trithioacetaldehyde.^{911a}

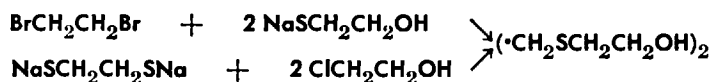
Sulfur dichloride can be added to vinyl chloride, vinylidene chloride, trichloroethylene, allyl chloride and styrene in the presence of ferric chloride.^{68a}

Sulfur mono- and dichlorides react with completely halogenated ethylenes, C_2F_4 , C_2ClF_3 , and C_2IF_3 . The crude disulfide, $(\text{ClCF}_2\text{CF}_2)_2\text{S}_2$, contains also monosulfide, trisulfide, tetrasulfide and pentasulfide. This agrees well with the observations on Levinstein mustard gas.¹¹³⁹

Perfluoropropyl sulfide, $\text{S}(\text{CF}_2\text{CF}_2\text{CF}_3)_2$, has been reported.⁶¹⁶

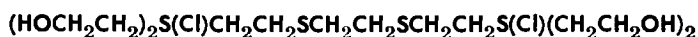
SESQUI-MUSTARD

From ethylene bromide and monothioethylene glycol, or from ethylene dimercaptan and ethylene chlorhydrin, ethylene bis- β -hydroxy ethyl sulfide is obtained.



This can be converted to the corresponding dichloride, $(\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl})_2$.^{114, 1200} This is comparable to mustard gas in vesicant action and in chemical reactivity. When injected, the effects on animals are quite different.^{318a} As has been stated before, the same compound is formed when mustard gas is heated.⁵²⁰ It is possible that the presence of some sesqui-mustard in the Levinstein product accounts for its high toxicity.⁹⁰⁰

Shaken with water at room temperature, sesqui-mustard is hydrolyzed.¹¹⁹⁶ Sulfonium complexes are intermediates. Along with the expected glycol, $(\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH})_2$, some of a higher glycol, $(\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH})_2$, is found.¹³¹⁹ In aqueous dioxane, the rate of hydrolysis follows the first-order equation.^{1123a} Sesqui-mustard unites with thiodiglycol to the sulfonium complex:



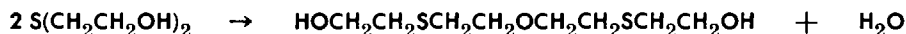
The sulfide-acid, $[\cdot\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}(\text{NH}_2)\text{COOH}]_2$, is produced by its reaction with cysteine.¹³¹⁹ The products with secondary amines are analogous to those from mustard gas.^{1123b} It yields dithiane when heated with hydrogen bromide in phenol.¹⁰¹ Oxidation converts it to the disulfoxide of which there are two forms, α , m. 180° and β , m. 152° , and then to the disulfone, m. $204\text{--}206^\circ$.^{1123a}

Mustard gas and sesqui-mustard have been converted to the sulfides, $\text{MeSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SMe}$ and $\text{MeSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SMe}$, by treatment with MeSNa . These derivatives are suitable for identification as they are nontoxic and can be distilled.⁹⁴¹

The fluorine analog of sesqui-mustard, $(\cdot\text{CH}_2\text{SCH}_2\text{CH}_2\text{F})_2$, has been prepared. It is a mobile liquid, devoid of vesicant properties.⁸⁸⁸

MUSTARD-T

Mustard-T, $\text{O}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl})_2$, has been made, starting with dichlorodiethyl ether and mercaptoethanol. It is a by-product in the preparation of mustard from thiodiglycol and hydrochloric acid. Two molecules of thiodiglycol are converted to the ether under the influence of the acid:



At higher temperatures, the amount formed becomes considerable. It is left as a residue when thiodiglycol mustard is distilled. Its reactions are similar to those of mustard. The diphenoxy derivative melts at 60° and its bis-tribromophenoxy derivative at 71° .^{1482a} Its hydrolysis proceeds through sulfonium complexes as intermediates.¹¹²²

HOMOLOGS

Trithiane with sulfur monochloride (70% yield), or with sulfur dichloride (100% yield), gives dichloromethyl sulfide, $\text{ClCH}_2\text{SCH}_2\text{Cl}$,^{142, 342, 911c} sulfoxide m. 40° .^{911c} It is one of the products when trithiane is chlorinated.^{389, 1397} It has been prepared also by the chlorination of methyl sulfide.^{451, 1154, 1174} This compound is comparatively inactive physiologically. It is toxic by intravenous injection, but its effects are radically different from those of mustard gas.^{318d} The rate of hydrolysis by water

is rapid but measurable.¹¹⁹⁶ On hydrolysis with hot water, there is a very bad odor which is supposed to be due to the transient presence of monomolecular thioformaldehyde. With methanol and alkali, the dimethyl ether, $\text{CH}_3\text{OCH}_2\text{SCH}_2\text{OCH}_3$, is formed.^{850b} With potassium cyanide in methanol, this may be obtained as a polymer.³⁴² The sulfone, $(\text{ClCH}_2)_2\text{SO}_2$, melts at 72° .^{850b} The corresponding bromosulfide, $(\text{BrCH}_2)_2\text{S}$, is rapidly hydrolyzed by water.¹¹⁹⁶

The corresponding selenium compound, $\text{Se}(\text{CH}_2\text{Cl})_2$, has been made by chlorinating trimethylene triselenide.²⁰⁰

The trichloromethyl sulfide, $\text{ClCH}_2\text{SCHCl}_2$, and the tetrachloromethyl sulfide, $\text{Cl}_2\text{CHSCHCl}_2$, are among the products when trithiane is chlorinated.^{203, 388, 565} The chlorination of methyl sulfide gives all possible chlorination products.^{451, 1397} These are listed in the properties. Hexachloromethyl selenide results from the chlorination of carbondiselenide.^{713.7}

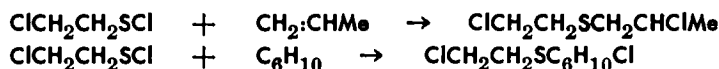
The perfluoromethyl sulfide, F_3CSCF_3 ,¹⁹⁵ and mixed chloro-fluoro sulfides¹³⁹⁷ have been prepared and their properties studied.

The addition of chloromethylsulfenyl chloride to ethylene gives a chloromethyl chloroethyl sulfide: ^{203, 388}



It can be added to other unsaturates.^{204.5, 205}

It has been noted in the treatment of the formation of mustard gas, that β -chloroethylsulfenyl chloride adds to ethylene. A similar reaction takes place with propylene and cyclohexene: ^{517, 522, 523}



The products are vesicant but much less so than mustard. The first of these has been prepared from the glycol.¹⁴⁶²

The isomeric α -chloroethanesulfenyl chloride, MeCHClSCI , has been added to unsaturates. With acetylene the product is $\text{MeCHClSCH}:\text{CHCl}$. Cyclohexene gives α -chloroethyl-2-chlorocyclohexane. Hydrogen chloride is lost from the addition products with styrene and cyclopentadiene, leaving β -chloro- β -phenylethyl vinyl sulfide and 4-chloro-cyclopentene-2-yl vinyl sulfide, respectively.^{202.3}

The γ,γ -dichloropropyl sulfide has been prepared from the corresponding glycol.^{110, 111a, 350a} The sulfone, $(\text{ClCH}_2\text{CH}_2\text{CH}_2)-$

SO₂, melts at 66°. The glycol was made from two starting materials, the chlorhydrin, HOCH₂CH₂CH₂Cl and γ -benzyloxypropyl chloride with sodium sulfide. The δ,δ' -dibenzyloxybutyl sulfide with fuming hydrobromic acid gave tetramethylene- δ -hydroxybutyl-sulfonium bromide.

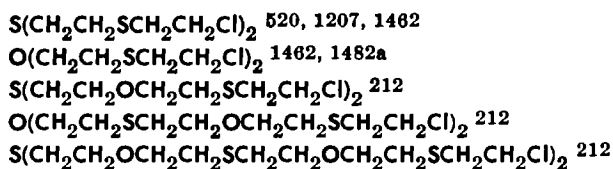
The reactivity of the chlorine in the sulfides S[(CH₂)_nCl]₂ is in the order $n = 1 > 2 > 3 < 4$. The chlorine in mustard gas is sixty times as reactive as that in the γ,γ' -dichloropropyl^{111b} sulfide.

Attempts have been made to prepare ClCH₂CH₂SCHMeCH₂Cl from the corresponding glycol,^{599, 1462} but it appears that isomerization takes place to give ClCH₂CH₂SCH₂CHClCH₃.⁵²³ This has been explained before.⁹

OTHER CHLOROSULFIDES

A group of dichlorides, ClCH₂CH₂SRSCH₂CH₂Cl, has been prepared in which R is methylene, propylene, trimethylene, α -methyltrimethylene, tetramethylene, pentamethylene, hexamethylene, octamethylene, nonamethylene, and decamethylene.^{350c, 530} 1,4-Butane-bis-sulfenyl chloride, ClS(CH₂)₄SCl, has been added to ethylene and to cyclohexene; the products are ClCH₂CH₂S(CH₂)₄SCH₂CH₂Cl and 2-ClC₆H₁₀S(CH₂)₄SC₆H₁₀-Cl-2.^{202,7}

Several long-chain dichlorides, having sulfur atoms in β -positions to the chlorine atoms and sulfide or ether linkages in the chain, have been prepared. All of them are more or less vesicant. Some of these are:



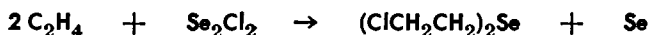
The chlorosulfides, CH₂(SCH₂CHClCH₂Cl)₂ and CH₂-[SCH(CH₂Cl)₂]₂, are from the dichloromercaptans and formaldehyde.¹⁰⁸² Chloral and hydrogen sulfide give S[CH(OH)CCl₃]₂.^{597.5, 853, 959, 1080, 1487}

From concentrated hydrochloric acid and the glycol, S[CH₂CH(CH₃)OH]₂, and from sulfur chloride and propylene, dimethyl mustard, S(CH₂CHClMe)₂, is obtained.^{303, 350a} From 2-butene and sulfur chloride, we get the tetramethyl mustard, (ClCHMe·CHMe)₂S. These have almost no vesicant action.

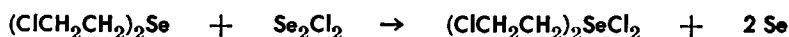
With isoamylene, the product appears to be the disulfide, $(\text{ClC}(\text{CH}_3)_2\cdot\text{CH}(\text{CH}_3)\text{S}\cdot)_2$, b_{11} 150–160°. From allyl chloride and sulfur monochloride the sulfide, $(\text{ClCH}_2\cdot\text{CHCl}\cdot\text{CH}_2)_2\text{S}$, is obtained at 100° and the disulfide, b_{11} 190°, at room temperature. With styrene the product is $(\text{ClCHPh}\cdot\text{CH}_2)_2\text{S}$.¹¹¹³ Allyl sulfide takes up bromine to form tetrabromopropyl sulfide, $(\text{BrCH}_2\text{CHBrCH}_2)_2\text{S}$.⁸⁹⁴ The sulfone, $(\text{BrCH}_2\text{CHBrCH}_2)_2\text{SO}_2$, m. 98–100°, is formed similarly from diallyl sulfone.^{850b} The addition of sulfur chloride to a polymerized olefin is said to give a surface-active agent after treatment with sodium carbonate.⁴⁸⁸ 2-Chlorocyclohexyl sulfide, said to be useful as an insecticide, results from the reaction of cyclohexene with sulfur dichloride.⁹⁹¹ Sulfur dichloride adds in two ways to butadiene to give $(\text{ClCH}_2\text{CH}:\text{CHCH}_2)_2\text{S}$ and $(\text{CH}_2:\text{CHCHClCH}_2)_2\text{S}$.^{1038.5} $\epsilon,\epsilon,\epsilon$ -Trichloroamyl sulfide, $(\text{Cl}_3\text{CCH}_2\text{CH}_2\text{CH}_2\text{CH}_2)_2\text{S}$, has been prepared from the chloride and sodium sulfide.^{705c} ζ -Chlorohexyl sulfide, $(\text{ClCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2)_2\text{S}$, is claimed to be effective against certain organisms.²⁸⁹ It causes nausea and asphyxiation.¹⁴²⁰

SELENIUM MUSTARD

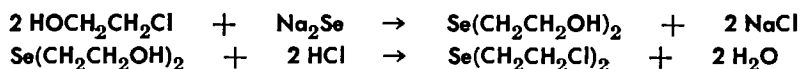
Ethylene reacts with selenium monochloride: ¹⁶³



The dichloroethyl selenide robs a further amount of the selenium chloride of its chlorine: ^{92, 102, 163, 617}



The chlorine on the selenium can be removed by treatment with potassium iodide. Bis(β -methoxyethyl) selenide, $\text{Se}(\text{CH}_2\text{CH}_2\text{OMe})_2$, has been made from the bromo-compound, $\text{Se}(\text{CH}_2\text{CH}_2\text{Br})_2$.¹²⁹² Selenium mustard can be made also by way of the selenodiglycol: ¹¹⁰⁴



The seleno-mustard, $(\text{ClCH}_2\text{CH}_2)_2\text{Se}$,²⁰⁷ is comparable to mustard gas in vesicant action.^{463, 617} The bromine analog, $(\text{BrCH}_2\text{CH}_2)_2\text{Se}$, is known. Propylene, butylene, and amylene react similarly with selenium monochloride.¹⁶³

MONOCHLORO-COMPOUNDS

These have been considered earlier in Chapter 4. All compounds that contain the group $\text{—SCH}_2\text{CH}_2\text{Cl}$ are more or less vesicant, but none of them approaches mustard in this respect.

NITROGEN MUSTARDS

These are not sulfur compounds but, on account of their close analogy to mustard gas, are mentioned here. When the hydrochlorides of the ethanol amines are treated with thionyl chloride they are converted to the hydrochlorides, $\text{ClCH}_2\text{CH}_2\text{NH}_2\cdot\text{HCl}$, m. 144° , $(\text{ClCH}_2\text{CH}_2)_2\text{NH}\cdot\text{HCl}$, and $(\text{ClCH}_2\text{CH}_2)_3\text{N}\cdot\text{HCl}$, m. $130\text{--}131^\circ$.¹⁴³¹ The free bases may be obtained from these by the addition of alkali in the presence of ether. The di- and tri-compounds, $\text{HN}(\text{CH}_2\text{CH}_2\text{Cl})_2$ and $\text{N}(\text{CH}_2\text{CH}_2\text{Cl})_3$, are strongly vesicant. The halogens in these are comparable in reactivity to those of $\text{ClCH}_2\text{CH}_2\text{SH}$ and $(\text{ClCH}_2\text{CH}_2)_2\text{S}$. With sodium alcoholates, phenates, and mercaptides, ethers and thioethers, $(\text{ROCH}_2\text{CH}_2)_2\text{NH}$, $(\text{RSCH}_2\text{CH}_2)_2\text{NH}$, and $(\text{PhOCH}_2\text{CH}_2)_2\text{NH}$, are formed.

Physiological Effects

TOXICITY

Breathing air containing 0.5 mg. per liter of mustard gas for 5 minutes or 0.01 mg. per liter for 8 hours will kill an animal.⁹¹⁹ It is fifteen times as toxic as chlorine.⁷⁷⁰ The effects of breathing mustard gas are cumulative; if the concentration is halved the time of exposure must be doubled. If c is the concentration in milligrams per liter and t is time in minutes, then

$$t = \frac{2000}{c}$$

This holds for rats.^{1209a} The lethal dose for a man is about 0.07 cc.¹⁴⁴⁹ By injection, water containing 650 mg. per liter is fatal to one rabbit out of three.²⁵

SENSITIVITY

Several forms of apparatus have been devised by means of which exposures of surfaces to accurately controlled concentrations of toxic vapors can be made^{435, 761, 1046b} and methods have

been developed for the study of effects on the skin.^{510, 1259a} Keratitis may be evident 10 to 20 years after injury.³⁷

Quantitative methods for determining the sensitivity have been devised and it is shown that some men are six hundred times as sensitive as others. About 3% are hypersensitive and about 30% comparatively resistant. On the average, Negroes are less sensitive than white men. Perspiration increases sensitivity.^{881, 920} Tests on 2728 men showed 4.9% supersensitive, affected by a concentration of 0.01%, 47% normal, responding to 0.1%, 42% resistant to this concentration and 6% unaffected by 1%.⁸⁵ A study of a smaller group showed similar wide variations of sensitivity.⁷⁶⁰

The time of exposure necessary to produce a positive reaction bears a definite relation to concentration and varies for different individuals.^{184, 1297} Treatment of the skin before exposure influences the results.^{329b} Repeated exposure increases the sensitivity.^{881, 968, 995, 1259b} This does not apply to a different skin area on the same person.¹³⁰⁵

The fluid content of the vesicles does not contain any vesicant.¹³³⁷

Rats under a week old are not affected by mustard gas on the skin.⁸⁴⁹ The rabbit and dog are less sensitive than man^{786b} while a horse is much more so.^{199, 881, 920} Guinea pigs and monkeys are more resistant.^{881, 920} As the species variations are not great the figures obtained with laboratory animals are probably nearly correct for men.⁹¹⁹

The eyes of man are extremely sensitive. Exposure of less than one hour to a concentration of 0.0005 mg. per liter will produce a reaction.¹¹⁵⁰ The effects on the eyes of man and of animals have been studied extensively.^{437, 492, 493, 494, 495, 496, 497, 535, 642, 681, 914, 931, 1100a, 1232, 1435}

TOXIC EFFECTS

The physiological effects of mustard gas have been reviewed.^{553, 1096, 1194, 1208} Sulfonium complexes appear to be involved.^{32, 553}

Mustard gas is destructive to any part of the body with which it comes in contact and causes more or less damage throughout the whole body from any point of application.⁵³¹ Its toxic effect is general, though it may result from a local application.^{329a, 475, 852, 1289} Its effects extend from the skin to the bone marrow.⁵⁸⁰

The distribution has been studied with the aid of radioactive sulfur^{52, 181} and its penetration compared with that of other vesicants.¹⁰²³ It is a protoplasmic poison and kills the cells. Its effects are similar to those of X-rays.⁵⁵³ Repeated application to the skin has the cumulative effect of a massive dose of X-rays.^{456b} The respiratory and glucolytic functions of the cells are affected.⁷²⁶ *In vivo* less than 10 gamma per gram of rat is toxic, but *in vitro* more than thirty times this amount per cubic centimeter is required. This indicates some specific effect, such as the poisoning of an enzyme.^{1030, 1089b} The eyes are the most sensitive to attack. Next are the lungs which are followed by the skin.¹⁴⁷⁶ Death usually results from pulmonary alterations^{877a} rather than from skin irritations. It acts on the upper respiratory tract and prevents the absorption of oxygen in the lungs.⁵³⁴ The danger to the lungs is lessened somewhat by absorption during its passage through the nose.²⁴⁷

It is slow acting; usually there are no symptoms for 4 or 5 hours and sometimes not for a day. This gives it time to be absorbed and carried to all parts of an organism. The delay may be apparent rather than real.¹³⁷ It affects even the marrow of the bones.^{879, 1031, 1073, 1074} This is important since by depleting the leucocytes in the circulation, it diminishes the ability to resist infection.⁸¹⁷ The lungs show edema and the intestines lesions. The blood pressure falls, the leucocytes increase at first, then decrease progressively and may disappear entirely. There is a fall in body temperature and progressive emaciation.⁹³⁵ The effect of the vapor on the epidermis of the skin and on the epithelium of the respiratory tract is escharotic; there is necrosis of the cells which may reach its height as late as 5 to 10 days after application.¹⁴³³ Many other disorders caused by mustard gas have been noted.^{246, 474, 1132, 1492} There are a number of recent studies.^{32, 136b, 241, 424, 456a, 874, 1209b}

The blood vessels are attacked, especially the capillaries, blood degeneration occurs and all metabolic processes are disturbed. The spleen and liver may be affected.¹⁴²³ The poison gains entrance to the blood through the injured epithelium and capillaries⁶²⁴ and damages the blood.^{1014e} As it acts through the blood, it is a general tissue poison.⁹⁰⁴ Its effects on the blood have been studied extensively.^{80, 273, 274a, 318b, 567a, 728, 774a, 811, 951, 1101, 1105, 1141}

Mustard gas painted on the skin of a cat affected the blood

cells; ⁴⁰¹ on the skin of a young rat it inhibited glycolysis within 5 minutes, but prolonged contact was required to stop respiration.⁸⁶ Application to skin of a white rat or guinea pig causes 30 to 40% loss of weight due to loss of appetite, increased oxygen consumption and to some other unknown factor.^{1371b} Rabbits after injection showed salivation, vomiting, diarrhea, convulsions, and slow and irregular heart action.⁸⁸¹ Intradermal injection into colored mice caused permanent graying similar to that produced by X-rays.¹⁸⁵ The effect on the thymus of a rat, injected with mustard, has been studied.^{274b} The effects on dogs have been studied extensively.^{60, 61, 273, 317, 424, 774b, 935}

Aqueous solutions of mustard gas deprive *E. coli* B organisms of their ability to divide without impairing their ability to form virus.^{638a}

Bee venom and mustard gas are superficially alike, but differ in their effects on the blood and urine.¹²⁸³ Mustard gas affects the production of adrenaline.⁹⁰⁹ The vesicant effect is observed only in man; animals show only subcutaneous edema.^{1371a}

On muscles the so-called Lundsgaard effect, inhibition of lactic acid formation, is produced.^{58a, 59, 64, 571}

The general effects of combat gases have been described.^{28, 305, 319, 551, 877b, 934, 945, 1252, 1412} The effects on soldiers at the front were: violent conjunctivitis with swelling of the eyelids, scorching of the air passages, inflammation of the skin, and the production of large areas of pigmentation.⁶³⁷ Those burned with mustard, show after the first week a diminution of the urine with higher acidity, less urea and chlorides excreted.⁶⁴¹ After 5 to 8 days the blood pressure was lowered and remained so for some time, so did the leucocyte count which was very low shortly before death in a soldier who died from bronchopneumonia.¹⁵²¹ In severe poisoning, the epithelial lining of the larynx and especially of the trachea beneath the vocal chords disappears very rapidly. The lesions give opportunities for infection.⁶¹³ Bronchopneumonia is a frequent cause of death.⁵⁵⁰ The amount of carbon dioxide exhaled is lowered sharply and comes back slowly.³ A soldier who had been gassed appeared normal, but became dizzy and faint with exercise.¹⁰⁸⁶

Low concentrations of mustard gas hemolyze red corpuscles *in vitro* by slow liberation of hydrochloric acid. This effect may be prevented by buffering.⁹⁰³ Yeast is poisoned by a selective

action on its reproductive functions, but the products of hydrolysis, hydrogen chloride and thiodiglycol, do not poison yeast.^{776a, 776c, 902} Mustard gas, either from thiodiglycol or from the Levinstein process, that has been distilled, inhibits the growth of yeast cells, but if the material is purified by recrystallization from alcohol it has scarcely any effect. Heating it to 85° for 12 hours restores the toxicity.¹⁴¹³ It reacts with horse-serum proteins at room temperature changing their immunological properties.^{124, 182d} Human serum treated with mustard gas delayed the healing of skin lesions in mice.¹⁹

The effects on marine organisms have been studied.⁸⁶⁰ From experiments with ameba the action seems to be on the cell surface.⁷⁸⁹ The addition of mustard gas to lipid preparations containing cholesterol increases the interfacial tension toward physiological saline and decreases the hydrophilic properties of the lipoids.⁷⁹⁰

The action of dilute mustard-gas vapor in the air on plants is slower than that of chlorine.^{590, 591} The changes produced on vegetation have been described.^{465, 979, 1071}

THEORY OF VESICANT ACTION

The mechanism of the action of mustard gas, according to Marshall, is (1) rapid penetration of the substance into the cell by virtue of its high lipid solubility; (2) hydrolysis inside of the cell to hydrochloric acid and thiodiglycol; and (3) destruction of the cell or interference with its functions by the hydrochloric acid.⁹¹⁹ This is the simplest and most generally accepted theory, but objections have been raised to it.³³ The fact that vesicant action and rate of acid liberation do not run parallel in a series of compounds is considered to be against this theory.¹⁰⁹⁰ There is no apparent relation between the chemical reactivity of the halogen atom and the vesicant action of various compounds.^{780a} The toxicities of mustard gas, thiodiglycol, and several of its esters on urease were determined with exact regulation of the pH and these compared with the rates of hydrolysis.¹¹⁹⁷ The effects on the cardiovascular system or on the bronchial musculature are not duplicated by thiodiglycol, the hydrolysis product.^{318a} The fact that mustard gas sulfone is vesicant but does not hydrolyze in water is urged against this theory.^{818b} It has been found that the hydrolysis of mustard gas is much

slower in physiological saline solution than in water.⁹⁸⁷ It is difficult to reconcile the slow action of mustard gas with its rapid hydrolysis in water solution. It may be protected from hydrolysis by being hidden in the lipoids or in the corpuscles of the blood. It may be that the unique toxic properties of mustard gas are due to a particular relationship between its solubility in water, its partition coefficient between water and lipoids, and its rate of hydrolysis. The theory does not tell the whole truth. Mustard gas certainly penetrates. It is distributed throughout the organism as mustard gas. It does kill cells wherever it goes. Thiodiglycol is not toxic. It does not get into the cell, and thus can do the cell no harm. If it were liberated inside of a cell it might have some effect.

Giving mustard gas to one of a cross-circulating pair of dogs kills both, but if the cross circulation is not begun until 30 to 60 minutes after the dosage the second dog is not affected. This is taken to indicate that death is not caused by a secondary toxin.^{136a}

It has been suggested that the toxicity is due to the formation of the sulfone,^{818a} but to prepare the sulfone in the laboratory requires the strongest oxidising agents. Mustard gas is oxidised slowly by air in a phosphate-buffered solution. It does not interfere with biological oxidative systems.¹⁴⁰⁵

Vesicants, in general, precipitate proteins, but mustard gas, the most irritant, has little of this precipitating power.⁶⁰⁶ As there are free amino groups in proteins, the reactions of mustard gas with amines^{350a, 831} and with amino acids, which make up proteins, have been investigated.^{78a, 182a, 350a, 473, 583a, 612, 831, 997, 1027, 1299, 1312, 1318, 1320, 1321} Experiments with proteins have shown that they are definitely altered by mustard gas. Some are made unfit for their normal functions. With complex substances, such as proteins, clean-cut reactions can not be expected.^{123, 180, 346, 568, 612, 639, 738, 777a, 1027, 1052, 1090, 1100b, 1332, 1491} Radioactive sulfur has been used in tests with proteins. The mustard gas that was fixed was not associated with the lipoids.¹⁰⁶¹ The sulfhydryl groups decreased, but this accounted for only one tenth of the amount of mustard taken up.^{78b}

Mustard gas is known to react readily with alkyl sulfides to form sulfonium compounds. Its reaction with methionine has been studied.^{1299, 1318, 1319, 1320}

Some of the complexes which mustard gas forms with heavy metal salts are mildly toxic to rats.⁷⁴⁹

It has been conjectured that histamine is in some way concerned in the physiological effects of mustard gas. It is known that it inactivates certain enzymes and it has been suggested that this is due to its reactions with groups on which such activities depend.^{65, 70, 639, 1052, 1090}

Mustard gas combines with the nucleic acids and is partially inactivated by folic acid.^{88b, 422} The effects on nucleic acids have been investigated extensively.^{38, 232, 233, 234, 567b, 796, 1371c, 1385}

For strong vesicant action, the chlorine atom must be in the β -position relative to the sulfur.^{104b, 350b, 780b} The halogen in this position is unusually reactive, particularly in alkaline solution. It has been found that mercaptans, in alkaline solution, react extremely rapidly with alkyl halides, even with those that are only moderately active. Thus sodium thiophenate reacts about one thousand times as fast with *n*-butyl bromide as does sodium phenate.¹¹³⁷ It seems likely that mustard gas would react promptly with any cysteine that it might encounter in the animal body. Cystine is a component of all proteins and the cystine-cysteine equilibrium is believed to be of vital importance in life processes. Any disturbance of this equilibrium must have serious consequences. These considerations have led to a study of the reactions of mustard gas with sulfhydryl compounds, particularly with those derived from proteins.^{63, 134, 573, 651} It has been found that the concentration of glutathione in the blood is reduced.^{650, 776d, 818d} The reaction with keratin has been considered.^{1089c}

Contravention

TREATMENT OF THOSE GASSED

Naturally much study has been given to the treatment of those exposed to mustard gas or injured by it. It is beyond the scope of this book to go into this subject in any detail, but a few references are given.^{28, 305, 377, 542, 551, 664, 844, 878, 1014c, 1194, 1252, 1436} Practical summaries of antidotes and palliatives have been published.^{1188, 1378}

There is little that can be done to destroy, or counteract, the material that has penetrated deeply, but if that which remains on the skin or near the surface can be taken care of, the injury will be less severe. Therefore, time is an important factor: The sooner a remedy is applied the less damage there will be. The

early application of hot water and soap is effective.¹¹³¹ Bleaching-powder solution is recommended.^{1244, 1419, 1424} Ice water reduces local injury to a rabbit's skin, probably on account of dilution and slowing of penetration.⁶⁰⁴ The application of a 1% solution of methylene blue in glycerol to a dog's skin 15 minutes after the application of mustard gas prevents subsequent necrosis.⁷⁵³ Hexamethylene tetramine has a neutralizing effect.²¹³ Spraying the affected skin with a solution of an aminophenol, or aromatic amino acid or aminosulfonic acid is claimed to be beneficial.¹³³⁶ Treatment with an organic solution of cysteine does some good.^{957b} Applying powdered activated charcoal has been found beneficial.¹²⁴⁴ It has been suggested that the nitrogen base used should be able to entrain the mustard mechanically as well as to react with it chemically.¹³³³ Exposure of affected animals to smokes containing salts of calcium and sodium is said to mitigate the effects.¹⁰⁶³ Dichloramides, such as dichloramine-T are said to render mustard gas innocuous,²⁸⁰ so do monochloramides.¹⁰⁹⁵ A solution of dichloramine-T and soda is effective.^{877c} For mustard gas conjunctivitis immediate irrigation with a 0.5 to 1% solution of dichloramine-T in chlorcosane followed by frequent irrigation with boric acid solution is advised.¹⁴³⁵ *o*-Aminothiophenol, dithiocarbamates, and 2,5-dimercaptothiodiazole have been found to be beneficial.⁴⁹⁷ Coating the skin with paraffin is said to be helpful.^{402, 1014b, 1367} The injection of benzyl cinnamate has been of service.⁷²³ Salves, ointments, and soaps containing bleach and other ingredients have been proposed.^{990, 1014d, 1376b, 1377} Moisture increases the effect of mustard gas on the skin. Therefore, water is to be avoided in ointments. Linseed and other oils are desirable.¹³⁰⁵ Sodium silicate hydrolyzes mustard gas readily and a 1% solution may be used on burns.⁸⁰⁷ Dilute solutions of sodium bicarbonate and of potassium permanganate have been used by intratracheal injections in treating rabbits.³²⁷ Aqueous solutions of potassium permanganate and of tannin are recommended as dressings.⁷¹⁰

With dogs, administering large doses of atropine prior to injection of mustard gas did not affect the mortality rate or survival time.^{318c}

Soldiers who have been gassed have been helped by breathing oxygen.^{81, 1086} The clinical and therapeutic histories of ten patients suffering from mustard gas poisoning have been given.³⁷⁵

PROTECTION

Since mustard gas attacks all parts of the body, protection against it is a serious matter. At 16° the vapor penetrates a layer of gauze 40 mm. thick in 24 hours.⁹⁸¹ Naturally much thought has been given to this problem and all manner of proposals have been made.¹²⁶⁴ The ideal material for clothing should be impervious to the vapors and nonabsorbent for the liquid, yet, for the comfort of the wearer, it must be permeable to air and water vapor. No such ideal material has been found. Leather allows the passage of air and water vapor but absorbs the vesicant. Rubber, which is commonly used for protection against other materials, is somewhat permeable to vesicant vapors,¹⁴⁴⁹ but impervious to air and moisture. A whole suit of it is too uncomfortable to wear, but rubber is used in masks.³⁵³

Goldbeater's skin has been suggested,¹⁶⁵ so have fabrics treated with glue or gelatin, which is then insolubilized,^{708, 949, 982, 1300} or with various plastics.^{43b, 132, 281, 333, 428, 739, 1102, 1167, 1301, 1441} Halogenated rubber and hardened latex have been proposed.^{397c} Lead dioxide and other oxidising agents may be incorporated.^{1048, 1440} Fabrics may be coated with bleaching materials.⁸⁸⁹ Fabric or leather may be impregnated with soaps of a bi- or trivalent metal and a fatty or resinous acid.³⁹⁹ Cellophane protects against mustard gas.^{786c} A garment of material of this sort has been recommended.¹³⁴⁶ A polyamide plastic, with which the vesicant might react, has been claimed.⁶³⁵ Curiously enough the addition of up to 5% of water to a fabric hastens the penetration of mustard gas, but more water prevents it.³⁰²

An impregnated porous woolen fabric is recommended.⁵⁸⁹ Garments of chlorinated wool give protection. Wool can absorb a considerable amount of chlorine without much deterioration. A part of this chlorine is available for the destruction of mustard gas.^{720, 1155}

An electrofilter did not precipitate mustard gas completely.¹²³⁴ A combination of sealing and decontaminating materials has been patented for making a room tight against poison gas.^{432, 1441} A phenol-formaldehyde resin is recommended for coating steel containers.⁶⁷⁹ A paint resistant to mustard gas has been described.¹²³³

The requirements for a protective ointment are given by

Williams.¹⁴⁶⁴ It should protect for 24 hours, not rub off easily, not irritate or produce unpleasant effects. It should retain a proper consistency under service conditions and at body temperature, be readily prepared from available materials, and not be too expensive. Various ointments have been proposed and tested against mustard gas under controlled conditions.^{397a, 603, 786a, 982, 1014a, 1464}

DECONTAMINATION ^{1166, 1194, 1376b, 1376c}

Where it can be applied, bleaching powder is generally recommended ^{368, 392, 394, 547, 1022, 1442, 1471} for the destruction of mustard gas. It is generally available, inexpensive, and effective. When it is applied directly to the liquid mustard the reaction is violent, even to inflammation. Bleaching powder is both an oxidising and a chlorinating agent, so may detoxify in either, or both of these ways. It has been used extensively on open ground and on streets. Lack of reactivity may be due to excessive dryness of the bleaching powder.⁸⁶⁴ A solution of sodium hypochlorite and sodium bicarbonate has been recommended.¹⁰¹⁹ Calcium hypochlorite is advised with emphasis on washing.^{877a} Chloramine and dichloramine-T have been recommended for the skin.¹⁴⁴² For cleaning containers, which have been used for transporting mustard gas, rinsing with chloramine solution and steaming out have been recommended.^{51, 1376a} Sodium sulfide, which reacts rapidly with mustard gas and with all substances containing active chlorine, has been suggested.¹⁰²² Calcium polysulfide, or lime-sulfur, which is so widely used for spraying trees, seems to merit a trial. It may require the addition of a mutual solvent.¹⁰⁸¹

Since mustard gas is chlorinated with great ease to products which are relatively harmless, the emphasis has been on chlorinating agents. The sting of the bee is due to the beta chlorine atom; its removal is efficient detoxification. More attention might well be given to the chlorine atoms. They can be removed by hydrolysis which is satisfactorily rapid at moderate temperatures if the contact is sufficient. As shown in the section on reactions, mustard gas undergoes many metathetical reactions with extreme ease. Reactions of alkyl halides with sodium-sulfur compounds, NaSR, are many times more rapid than with the corresponding sodium-oxygen compounds, NaOR. There are diffi-

culties in the way, sodium and calcium sulfides and polysulfides react well with mustard gas, but it is difficult to get them into the proper contact. A sodium mercaptide reacts well, but is readily hydrolyzed and oxidised.

Fire has been considered for open ground.¹³³⁰ This is certainly effective, but there is danger that some of the material may be volatilized before it is burned. The effects of mustard gas on water works has been considered.^{231, 1314, 1481} Hydrolysis takes care of mustard gas dissolved in water, rapidly at a high temperature, or slowly at a low,¹³⁷⁵ but on account of its high density and low solubility a puddle of it may remain under water for a long time. Garments may be boiled or autoclaved with water,^{388, 883, 1376d} The action of water is greatly accelerated by the emulsifying effect of soaps.^{1467a} A chlorinated soap has been patented.¹²¹² Leather has been a problem.^{30, 36, 865} As paper would be injured by drastic treatments, documents can be decontaminated by exposure to ammonia gas for several days in closed containers.¹⁴⁹⁷

The protection and poisoning of foodstuffs have been studied.^{752, 866b, 1184, 1248, 1438, 1439} Flour may remain dangerous for a month at 16°. Sausages in cellophane skins withstand the gas for many days.⁶¹⁴

Extensive experiments have been made on the absorption of mustard gas by building materials. Asphalt roofing paper proved to be particularly resistant to penetration; roofing slate was found to be absorbent.¹³⁷³

Therapeutic Uses

There are reviews of this subject.^{62, 553, 870}

Mustard gas has been applied *in vitro* to heteroplastic transplanted tumors.¹²⁶⁹ The development of tumors from carcinogenic tars is inhibited by this substance. This effect seems to be due to some local action on the tissues, causing them to become resistant to the action of the tar.^{121, 122, 1417} When fresh, mustard gas is said to have no effect, but it does have a marked effect during its slow decomposition.³⁶⁴ There is considerable evidence that it hinders the growth of carcinoma.^{58c, 939, 1266, 1267, 1270} It inhibited the growth and caused regression of mice lymphoma.^{88a} Synthetic folic acid counteracts this effect.^{88b} Injection of a dilute solution into tumors has shown promising

results.^{4, 743} The glucolysis of minced sarcoma tissue is reduced more than its respiration.¹²² The cyclohexyl analog has shown the same effect on tumors as mustard gas.⁵⁹⁷ However, mustard gas is said to be carcinogenic.⁶⁴⁶

Mustard gas appears to be particularly effective against viruses.^{638c, 640, 1198} Its addition to brain suspensions from animals infected with fixed virus (rabies) gave a high-quality vaccine. Vaccines, effective against several diseases, have been prepared with its aid.¹³⁷² A study has been made of serums obtained from dogs that had been treated with mustard gas.⁵⁶⁸

Mustard gas in 25% glycerol is bactericidal toward virulent human tubercle bacilli *in vitro*.³²¹ It has an inhibitory effect on the development of pulmonary tuberculosis in rabbits.⁷⁹⁹ Mustard gas inhibits the action of urease.^{472, 583b, 868} In some cases, it causes the growth of hair.^{481, 1348} It counteracts the effects of the injection of sheep's blood into rabbits and guinea pigs.^{320, 625} It slows down, or inhibits, mitosis.^{338a, 776a, 776b, 776e, 918} Chloroethyl butyl sulfide, $\text{BuSCH}_2\text{CH}_2\text{Cl}$, has the same effect.^{776e}

Mutations are produced by mustard gas.^{39, 41, 338b, 544, 593, 653, 674, 731, 1365} There are only a few compounds that do this and all of them are vesicants closely related to mustard gas: $\text{O}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl})_2$, $\text{N}(\text{CH}_2\text{CH}_2\text{Cl})_3$, and $\text{MeN}(\text{CH}_2\text{CH}_2\text{Cl})_2$.^{39, 41, 42} The half-mustard, $\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$ is active.⁴⁰ The monochlorides, $\text{EtSCH}_2\text{CH}_2\text{Cl}$,⁸⁷³ $\text{BuSCH}_2\text{CH}_2\text{Cl}$,^{40, 1327a} and $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{Cl}$,^{1327a} show decided effects, greater than the aromatic chloride, $\text{PhSCH}_2\text{CH}_2\text{Cl}$.^{1327a, 1327b} A polychlorocyclohexyl sulfide is also active.²⁶⁸

Adsorbed on charcoal, mustard gas has been applied as a dust to plants; bean beetles were killed without injury to the plant.⁹¹⁶

USE IN WARFARE

Only a few brief statements can be made here on this subject which has been discussed at length in many articles and in books on chemical warfare. References to some of these are given in the introduction to this section.

In World War I¹¹¹⁹ (1917–1918), mustard gas was put over chiefly in shells. According to the force of the explosion and other circumstances it is scattered in droplets varying from the size of rain drops to a fine mist, barely visible in damp weather and not at all in dry. This is effective for 6 hours in open country

and 12 to 24 hours in places protected from wind and sun. Field tests have shown that a 12 mile per hour wind passing over a mustardized area may carry a concentration of 0.07 mg. per liter as far as 1000 yards. This is sufficient to cause death in 30 minutes. The amount carried by the wind will naturally depend on the amount of material on the ground and the temperature. Mustard gas is readily absorbed by clothing; there were cases in which the occupants of a dugout were made ill by that which was carried in on the clothing of a single soldier who was ignorant of its presence. On account of its persistence, mustard gas cannot be used on ground which is to be occupied within a short time. An area which has been drenched with it may be unsafe for days.

Pure mustard gas melts at 14.5°C. or 57.9°F. The impure material, such as is used, melts lower than this but is solid in cold weather. As the ballistics of a shell change when a liquid filling solidifies, enough of a solvent is usually added to keep it liquid at such temperatures as are likely to be encountered. The Germans and French added 10 to 25% of a volatile solvent, such as carbon tetrachloride, chlorobenzene, or nitrobenzene. The Americans used chlorpicrin which has some toxicity as well as solvent effect. It has been generally assumed that the evaporation of a volatile solvent aids in the evaporation of the mustard gas, but the vaporization of the more volatile liquid uses up a considerable amount of heat which might have been used to volatilize the other constituent. This fact seems to have been overlooked.

The fact that mustard gas is harmful on long exposure, in concentrations too small to be detected by its odor, makes it all the more dangerous. Its slowness of action is a detriment; men may carry on effectively in a toxic concentration and win a battle. They may go to the hospital the next day, but the victory is still theirs. Of the 160,970 gas casualties admitted to British clearing stations from July 21, 1917, to Nov. 23, 1918, 77% were due to mustard gas. Of the 160,970 casualties only 1,859 or 1.15% died. Of the 2,039,329 men of the A.E.F. who reported in France there were 70,552 gas casualties of whom 1,421 or 2.01% died. There were 187,786 hit by shot and shell of whom 34,249 died on the field and 12,470 in hospitals or 46,719 in all. Thus 24.9% of those hit died as compared with 2% of those gassed. Much has been said and written about the inhumanity

of the use of poison gas but ninety-eight out of every hundred injured by it came out of the hospitals. Some bore scars, but none had arms or legs missing.⁵⁴⁹

Altogether about 12,000 tons of mustard gas used in World War I caused about 400,000 casualties or one casualty for sixty pounds. If 2% of these died it took 3,000 pounds to kill a man. This compared with 20 mg., the minimum lethal dose, shows that most of it was wasted.

CHAPTER 6.

Sulfides Containing Other Substituents

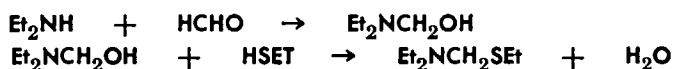
Sulfides containing carboxyl groups are to be found in the chapter on sulfide acids in Volume III. Those having other substituents are discussed in this chapter.*

Aminosulfides



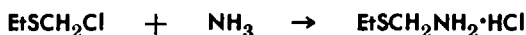
FORMATION

Aminosulfides in which $n = 1$ are a special class both as to their formation and reactions. They are from an amine, formaldehyde, and a mercaptan. Those in which the nitrogen is fully alkylated are the most stable. Diethylamine is dropped into 35% formaldehyde, with cooling, and mercaptan is added: ⁸⁹⁵



This is a variation of the Mannich reaction.

These compounds may be obtained, the other way around, by the reaction of a chloromethyl sulfide on ammonia or on an amine: ^{732b}



* As for the two preceding chapters the references are to be found in the "Bibliography" at the end of Chapter 7.

α -Chloroalkyl sulfides, such as EtSCH_2RCl , react similarly.^{732b}

Thiophenol condenses with hexamethylene tetramine to $\text{N}(\text{CH}_2\text{SPh})_3$. Substituted phenols do likewise.³⁸⁷

These aminosulfides can be converted to quaternary ammonium salts by the addition of an alkyl halide: ¹¹⁶⁵



These salts may also be obtained by the addition of a chloromethyl alkyl sulfide to a trialkylamine: ⁶⁹⁹

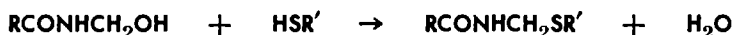


The melting points of some of these salts are in Table 4.7 in Chapter 7 on the physical properties.

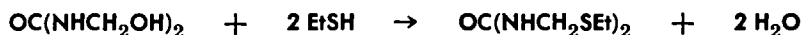
The pharmacological properties have been studied.^{685, 1165} Compounds containing higher alkyls, such as dodecyl, are claimed as fungicides,^{694a, 1366} bactericides,^{699, 1366} and wetting agents.¹³⁶⁶ Some with more complicated groups are mentioned as accelerators for vulcanization.^{735b}

Linear polyamides may be treated with formaldehyde and a mercaptan in the presence of a catalyst.²⁴⁵

Unsubstituted aminosulfides, RSCH_2NH_2 , are converted into isocyanates, RSCH_2NCO , by phosgene. These are said to make fabrics water repellant.⁷⁴² A methylolamide reacts with a mercaptan: ⁴⁶



Dimethylolurea reacts with mercaptans to form bis(alkylmercaptomethyl) ureas:

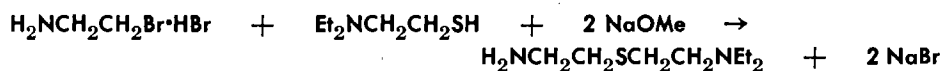


Half-way compounds, $\text{MeNHCONHCH}_2\text{SEt}$, and $\text{EtNHCONHCH}_2\text{SEt}$, are obtained from monoalkylureas. $\text{OC}(\text{NHCH}_2\text{SEt})_2$ is rhombic.⁷⁴⁴

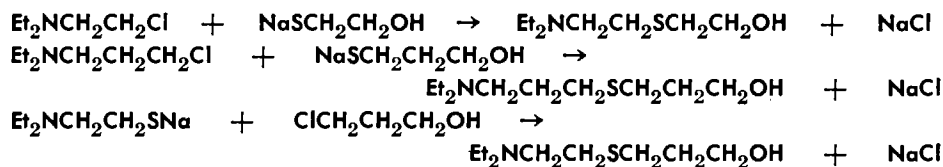
The aminosulfides, $\text{H}_2\text{N}(\text{CH}_2)_n\text{SR}$, in which n equals or exceeds two can be prepared by regular synthetic methods. Thus, $\text{C}_6\text{H}_4(\text{CO})_2\text{N}(\text{CH}_2)_n\text{Br}$, made from phthalimide, is caused to react with a sodium mercaptide and the product hydrolyzed to $\text{H}_2\text{N}(\text{CH}_2)_n\text{SR}$. β -Aminoethylethyl ¹²³⁹ and β -aminoethyl phenyl ^{527a} sulfides, $\text{H}_2\text{NCH}_2\text{CH}_2\text{SEt}$, and $\text{H}_2\text{NCH}_2\text{CH}_2\text{SPh}$, were made in this way. Or, the other way around, the bromoethyl-

phthalimide, $\text{C}_6\text{H}_4(\text{CO})_2\text{NCH}_2\text{CH}_2\text{Br}$, is caused to react with potassium xanthate and the product hydrolyzed to the amino-mercaptan, $\text{H}_2\text{NCH}_2\text{CH}_2\text{SH}$, which may then react with an alkyl halide to give an aminoalkyl sulfide.^{527b, 1020} Phthalimidoethyl thioformal has been hydrolyzed to the aminoethyl thioformal, $(\text{H}_2\text{NCH}_2\text{CH}_2\text{S})_2\text{CH}_2$.⁹⁶⁷ Aminoethyl sulfide, $(\text{H}_2\text{NCH}_2\text{CH}_2)_2\text{S}$, has been prepared by the phthalimide method.^{526b, 526c}

Mono-, di-, and triethanolamines, which have become available in recent years, can be converted to the chlorides, $\text{H}_2\text{NCH}_2\text{CH}_2\text{Cl}$,^{1265, 1431} $\text{HN}(\text{CH}_2\text{CH}_2\text{Cl})_2$, and $\text{N}(\text{CH}_2\text{CH}_2\text{Cl})_3$.¹⁴³¹ These "nitrogen mustards" have been mentioned in connection with mustard gas. The bromide, $\text{NH}_2\text{CH}_2\text{CH}_2\text{Br}$, can be made from ethanolamine and hydrobromic acid.²⁰¹ All of these react well with mercaptides. Since the free aminoalkyl halides are not stable, they are handled and stored as their salts. The salt may be mixed with the mercaptan and enough alkali added to form the mercaptide and to set free the amine:²⁹⁹



Dialkylaminoalkyl sulfides can be prepared either from a dialkylaminoalkyl halide^{115, 157, 301, 556b, 563, 1076, 1085, 1182, 1206} and a mercaptide or from a dialkylaminoalkyl mercaptan and an alkyl halide:^{126, 299, 301, 564, 827}

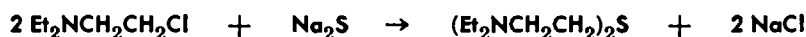


2-Chloroquinoline reacts satisfactorily with a mercaptan to give the sulfide.^{555, 703} Thioammeline can be alkylated by conventional methods.^{694d}

Benzhydryl mercaptan and diethylaminopropylene oxide give 3-diethylamino-2-hydroxypropyl benzhydryl sulfide, $\text{Ph}_2\text{CH-SCH}_2\text{CH}(\text{OH})\text{CH}_2\text{NEt}_2$.¹⁶⁹ *p*-Chlorothiophenol and epichlorohydrin give 4-chlorophenylmercaptopropylene oxide, which dibutylamine changes to 3-dibutylamino-2-hydroxypropyl chlorophenyl sulfide, $\text{ClC}_6\text{H}_4\text{SCH}_2\text{CH}(\text{OH})\text{CH}_2\text{NBu}_2$.¹⁸³ An amino-mercaptan may react with styrene oxide, or propylene oxide.

With *o*-aminothiophenol the products are: $\text{H}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{CH}(\text{OH})\text{Ph}$ and $\text{H}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{CH}(\text{OH})\text{Me}$.⁵¹⁵

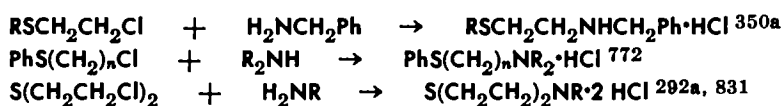
Symmetrical dialkylaminoalkyl sulfides can be made from the halide and alkali sulfide: 14, 311, 562, 1369



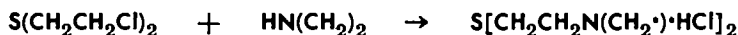
The sulfide, $\text{S}(\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2)_2$, was made from the phthalimidopropyl bromide.⁸³⁶

The amino group may be protected in other ways. From *N*-chloroamylbenzamide, $\text{PhCONH}(\text{CH}_2)_5\text{Cl}$, and sodium sulfide the sulfide has been made and hydrolyzed to the aminoamyl sulfide, $(\text{NH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2)_2\text{S}$.⁴⁷⁷

As has been discussed more fully earlier in this volume, chloroalkyl sulfides react with amines in the presence of sodium acetate or of a base:

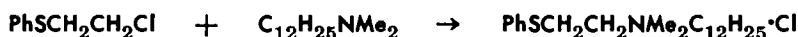


Ethylene imine reacts as a dialkylamine: 131

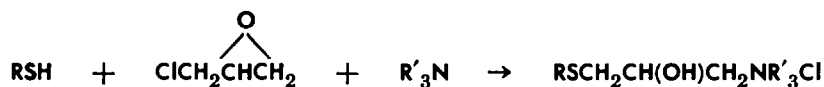


The reaction of *o*-nitrobenzenesulfene chloride with dimethyl-*p*-toluidine gives 2-nitro-2'-dimethylamino-5'-methyl-diphenyl sulfide.^{433a}

The addition of a chlorosulfide to a tertiary amine gives a quaternary ammonium salt: 1338



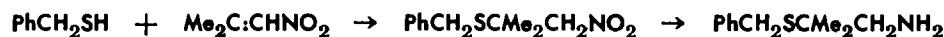
A mercaptan, epichlorhydrin, and a tertiary amine combine: 1058a



A mercaptan may be added to a basically substituted acetylene: 490, 692b, 700

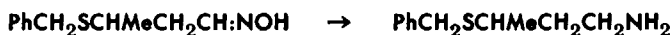


The addition product with an unsaturated nitro compound may be reduced: 328



Many aminosulfides have been prepared by the reduction of the corresponding nitro compound.^{8, 179, 228b, 263, 300a, 452, 454a, 471, 509, 556a, 725, 734b, 754, 803a, 992, 1041, 1092, 1129, 1223, 1307, 1339, 1349, 1515b, 1519}

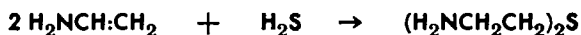
Reduction and deacetylation of *p*-acetylaminophenacyl *p*-nitrophenyl sulfide, $\text{AcNHC}_6\text{H}_4\text{COCH}_2\text{SC}_6\text{H}_4\text{NO}_2$, gives a diamino-sulfide.⁴⁵⁵ An aminoquinoline alkyl sulfide can be made by reducing the corresponding nitro compound.⁹²⁶ Aminopyridine alkyl sulfides have been made in this way.¹³⁵⁵ The oxime of a sulfide-aldehyde can be reduced to an amine:²⁶⁵



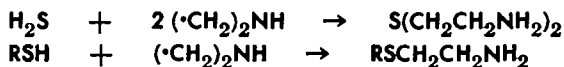
A sulfide nitrile may be reduced by sodium in alcohol: ^{10c, 11, 264, 1242}



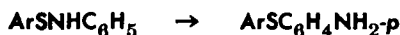
Vinyl amine reacts vigorously with hydrogen sulfide: ^{528, 880}



A method of synthesis which gives products in which the sulfur and nitrogen atoms are on adjacent carbons is the reaction of ethylene imine with hydrogen sulfide or with a mercaptan: ^{84, 120, 159, 946b, 978, 1010, 1028}



Aminoaryl sulfides are formed in the rearrangement of sulfen-anilides: ^{96, 734c, 996a, 996b}



These have been discussed in Chapter 3 of Volume I.

The reaction of sodamide with methyl *o*-bromophenyl sulfide gives the meta amino derivative instead of the expected ortho compound: ^{561, 923}



Aminothiophenols,^{820.5, 1049} acetylaminothiophenols,^{1457, 1514} mercaptoquinolines,¹³⁶⁸ and mercaptoimidazoles⁷³⁶ are alkylated by the usual methods. 2-Chloro-5-nitropyridine and potassium hydrosulfide give 5,5'-dinitro-2,2'-dipyridyl sulfide.¹³⁵³ The sulfides, 2,5-(PhSO_2NH)₂ $\text{C}_6\text{H}_3\text{SR}$, have been made by alkylating the mercaptan.⁶

Both *o,o'*-diaminodiphenyl sulfide¹⁰⁸⁴ and *p,p'*-diaminodiphenyl sulfide are prepared by heating aniline with sulfur.^{663, 952} Substituted anilines react in the same way.^{158, 335a, 335b, 952, 1399} Litharge may be added to take care of the hydrogen sulfide.^{655.5, 996a, 1399} The same amino sulfides are obtained by treating the aromatic amines with sulfur monochloride.^{599.5, 952, 1236} This reaction is carried out in solution at a low temperature. Diphenylamine and sulfur, heated together, give phenothiazine.¹²⁹ A sulfonium chloride, $(\text{Me}_2\text{NC}_6\text{H}_4)_3\text{SCl}$, has been obtained starting with dimethylaniline and thionylchloride.⁹⁶¹

Heating benzeneselenonic acid with aniline gives the aminoselenide, $\text{PhSeC}_6\text{H}_4\text{NH}_2$ -*p*.⁵³⁷ Other aminoselenides have been made by the reduction of the nitro derivative⁷⁵⁵ or from the selenocyanates.¹¹⁴⁵ Selenyl chloride reacts with dialkylanilines to give *p,p'*-tetraalkyldiaminodiphenyl selenides.⁵⁷⁰

REACTIONS

The aminosulfides undergo the reactions which are appropriate to sulfides and to amines, insofar as the two do not conflict. They may be converted into guanido compounds.^{727, 751, 1249} They may be oxidised to sulfoxides.⁷⁵¹ Usually the amino group is protected by acetylation.^{1510a, 1518} The sulfones may be obtained by further oxidation.⁵⁸² Another way to make the sulfone is the reaction of an aminobenzene sulfinic acid with an alkyl halide.^{454b} With cyanogen chloride, they give cyanamides.^{426, 620} An acyl-aminosulfide can be reduced to an alkylamino sulfide by lithium aluminum hydride.^{793b}

Aminosulfides condense with aldehydes and quinones.^{1510b} The condensation products of aldehydes with the diaminosulfides, $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NH}_2$,^{306, 412, 413} $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{NH}_2$,^{411a} $\text{CH}_2(\text{CH}_2\text{SCH}_2\text{CH}_2\text{NH}_2)_2$,^{411a} $\text{H}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NH}_2$,^{411a} and $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NH}_2$,^{411b} form complexes with the heavy metals, particularly cobalt. Methyl *p*-aminophenyl sulfide and paraldehyde condense to 6-methyl-mercaptoquinoline.¹³⁵⁴

Certain aminophenyl sulfides rearrange into mercaptodiphenylamines under the influence of alkali.^{433a, 433b}

The resolution of β -aminoethyl sulfide-platinum chloride complex is regarded as important in that it demonstrates the reality of the coordinate bond.⁹¹⁰

Diazotized *p*-methylmercaptoaniline, treated with mercuric chloride and then with copper, is converted to *p*-methylmercapto-phenylmercury, $(\text{MeSC}_6\text{H}_4)_2\text{Hg}$.⁴²⁷ Diazotized aminosulfides appear to generate free radicals in aqueous solution.¹²³⁰ A small yield of dibenzothiophene is formed in the decomposition of the diazonium salt from phenyl *o*-aminophenyl sulfide.³⁷⁰

In an extensive study of the effect of sulfur on the colors of dyes, many ortho,⁴⁸⁵ meta,¹⁰⁶⁹ and para¹⁴²⁶ aminophenyl sulfides, $\text{RSC}_6\text{H}_4\text{NH}_2$, were prepared. These were diazotized and coupled with several intermediates. The dyes were compared with the corresponding ones containing the MeO — group. In all cases, the sulfur had a decided bathochromic effect. This has been confirmed by others.^{656b}

β,β' -Diaminodiethyl sulfide, $\text{S}(\text{CH}_2\text{CH}_2\text{NH}_2)_2$, condenses with acetanhydride to form 2-methylthiazoline.^{526b} Sulfonamides have been made from this amine with the chlorides of several substituted benzenesulfonic acids.⁸³⁷

The absorption spectra of several acetylaminophenyl sulfides have been recorded.⁹⁰⁸

USES

The toxicity of bis(aminoethyl) sulfide has been studied.⁸⁸⁰ Diethylaminoethyl alkyl sulfides, such as $\text{Et}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl}$, $\text{Et}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$, and $\text{Et}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NH}_2$, are used in the synthesis of pharmaceuticals.^{296, 297, 298, 478, 692a, 701, 969} The triphenylmethyl compound, $\text{Ph}_3\text{CSCH}_2\text{CH}_2\text{NMe}_2$, is an antihistamine.¹¹⁸² Some thiophene derivatives, $3\text{-C}_4\text{H}_3\text{S-S}(\text{CH}_2)_n\text{NR}_2$, are antihistamines and anesthetics.⁶⁷⁵ Local anesthetics containing alkylmercapto groups have been prepared.⁸²¹

p-Aminophenyl sulfide, $(\text{H}_2\text{NC}_6\text{H}_4)_2\text{S}$, has fungicidal properties.⁵⁷⁶ The Schiff's bases obtained from it have bactericidal activity.¹¹⁴² *p*-Chlorophenylmercaptoaniline, *p*- $\text{ClC}_6\text{H}_4\text{SC}_6\text{H}_4\text{NH}_2$, is bacteriostatic for some strains of tubercle bacillus.⁴⁸⁹ Some derivatives of quinoline sulfides have been of interest.^{300b, 1451} The quaternary ammonium salt, $\text{S}(\text{CH}_2\text{CH}_2\text{NMe}_2\text{C}_{12}\text{H}_{25})_2\text{Cl}_2$, has been tested for germicidal activity.¹³³⁸

Aminoalkyl sulfides have been used in making vulcanization accelerators and insecticides.^{927, 928} Some quaternary salts are recommended as detergents.^{1058a} Certain substituted aminoalkyl sulfides are said to be useful as emulsifiers in the polymerization

of butadiene mixtures.¹⁰⁷² Diazothioethers have been used in emulsion polymerization.⁷⁹⁷

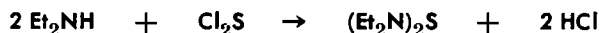
Sulfides derived from thioammeline are said to be useful in plastics, synthetic resins, and other products.^{216, 219, 336, 337, 1168, 1498, 1499} Those containing a pyrimidine ring are mentioned in some of these patents.^{336, 337}

A basic ion-exchange resin is obtained by condensing tetra-ethylenepentamine with 2,3-epoxypropyl sulfide.⁴⁰³

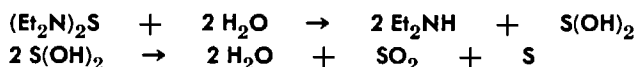
Thioamines

These do not really belong here, since the sulfur is not joined directly to carbon, but they are sulfides and do contain alkyls.

A secondary amine and sulfur dichloride react in ligroin solution:



This boils at 190°, with decomposition, or at 85–8° at 15 mm. Dipropyl, di-*i*-butyl, and di-*i*-amyl amines react similarly. The products may be regarded as alkylated amides of the unstable acid, S(OH)₂, into which they are hydrolyzed:^{840, 960b}

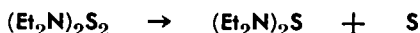


This synthesis may be carried out in dry ether as well as in ligroin.^{138, 960b}

A dialkylamine reacts also with sulfur monochloride: ^{138, 960b, 964, 1291, 1496}



(Me₂N)₂S₂, b₂₂ 82–3°; (Et₂N)₂S₂, b₂₉ 137–8°, piperidine disulfide, m. 64°, morpholine disulfide, m. 125°, ⁹⁶⁴ (Pr₂N)₂S₂, yellow oil, (*i*-Bu₂N)₂S₂, m. 31°, and [(PhCH₂)₂N]₂S₂, m. 79°. ^{848a} On heating, the disulfide is converted to the monosulfide: ^{960b}



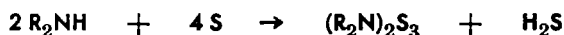
It has been known for some time that primary amines react with sulfur monochloride,^{885, 964} but only recently have definite products been isolated. The one from ethylamine melts at 35° and appears to be the cyclic dimer of EtN·S·S.^{848a}

The reaction of sulfur monochloride with dialkylamines can

be carried out in aqueous solution or suspension^{135, 705a, 707} or in an organic solvent such as benzene, enough water being added to dissolve the amine hydrochloride.^{735a} Certain of the products, so obtained, are claimed as vulcanization accelerators.^{1390.5} Aniline,^{303, 1455.5} diphenyl amine,^{670.5, 1060.5} and di- β -naphthyl amine^{821.5} react with sulfur monochloride to give products of uncertain constitution.

The Raman spectra of $\text{Me}_2\text{NSNMe}_2$, $\text{Me}_2\text{NSONMe}_2$, and $\text{Me}_2\text{NS}_2\text{NMe}_2$ have been studied.⁵⁷²

A secondary amine and sulfur react if lead oxide is present to take care of the hydrogen sulfide:



Some amines can be made to take up another atom of sulfur. The trisulfide from dimethylamine melts at -28° , that from the diethyl is an oil. Piperidine gives a trisulfide, m. 74° , and a tetrasulfide, m. 78° .^{848a}

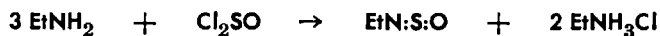
Amine monosulfides are converted to trisulfides by treatment with sulfurous acid, and the disulfides to tetrasulfides by the same means.^{696, 697}

Morpholine and piperidine disulfides and polysulfides are anti-fogging agents for silver halide emulsions.¹⁰⁰⁹ A mixture of N,N'-dithiodiethylamine and barium trithiocarbonate has been recommended for the vulcanization of rubber.^{735c} Various amine sulfides are claimed to be useful in vulcanization.^{95, 139a, 601, 1285, 1390}

Sulfide, or disulfide, amides, whose structures are somewhat uncertain, are obtained by treating amides with sulfur monochloride.^{1024, 1025, 1026}

Thionyl Amines

A primary amine reacts with thionyl chloride in anhydrous ether: ^{960a}

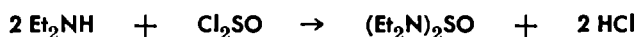


The thionyl amine remains in the ether while the amine hydrochloride separates out. Several compounds of this class have been described: MeN:S:O , b. 58 to 59° , EtN:S:O , b. 70 to 73° , PrN:S:O , b. 104° , $i\text{-BuN:S:O}$, b. 116° , AmN:S:O , b.₅₀ 85° ,⁹⁶⁵ $\text{CH}_2:\text{CHCH}_2\text{CH}_2\text{CHMeN:S:O}$, b. 156 to 158° , d₁₅ 0.9986 ,⁹⁶³

$\text{Et}_2\text{N}\cdot\text{N}_2\text{S}_2\text{O}$, b_{20} 73° , which is hydrolyzed by water,⁹⁶⁵
 $\text{O}:\text{S}:\text{NCH}_2\text{CH}_2\text{N}:\text{S}:\text{O}$, b_{25} 100° , m. 5.5° , and $\text{CH}_2(\cdot\text{CH}_2\text{N}:\text{S}:\text{O})_2$,
 b_{26} 117° .⁹⁶²

A study has been made of the dipole moments of these compounds in the hope of gaining more information as to their configuration.⁷³⁰

Two molecules of a secondary amine react with one molecule of thionyl chloride:

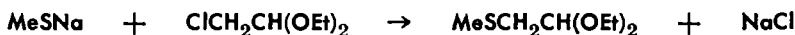


This boils at 118° at 27 to 28 mm. pressure and has d_{15} 0.9854.^{960b}
 It is a tetraalkylamide of sulfurous acid.

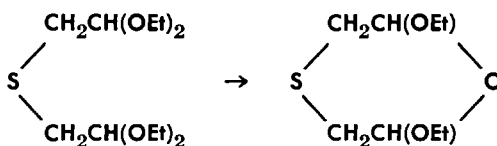
Strictly speaking, these compounds of sulfurous acid do not belong here but are included for comparison.

Sulfide-Aldehydes

A haloacetal reacts with a mercaptide: 48, 536, 1021, 1074.5, 1074.7, 1075.5



With sodium sulfide the product is the sulfide, $\text{S}[\text{CH}_2\text{CH}(\text{OEt})_2]_2$.⁴⁶⁹ The dialdehyde, $\text{S}(\text{CH}_2\text{CHO})_2$, has been obtained. In aqueous solution, it exists apparently in equilibrium with 3,5-dihydroxythioxane.^{304, 980} The *p*-nitrophenyl compound, $\text{O}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{CH}(\text{OEt})_2$, has been made from *p*-nitrothiophenol.¹⁰⁵⁶ Several other sulfide acetals have been reported: $\text{PhCH}_2\text{SCH}_2\text{CH}(\text{OEt})_2$ ^{645, 690} and $\text{PhSCH}_2\text{CH}(\text{OMe})_2$.¹¹⁴⁰ A sulfide-acetal is converted by hydrogen chloride into a thioxane derivative:²⁹³



Methylmercaptopropionaldehyde, $\text{MeSCH}_2\text{CH}_2\text{CHO}$, has been prepared from the acetal, $\text{MeSCH}_2\text{CH}_2\text{CH}(\text{OEt})_2$. This aldehyde and the corresponding alcohol, $\text{MeSCH}_2\text{CH}_2\text{CH}_2\text{OH}$, are partly responsible for the odor of soy sauce.^{10b} The aldehyde has become important as an intermediate in the synthesis of methionine and will be mentioned again in the chapter on methionine. Its

direct preparation from methyl mercaptan and acrolein has been found to be possible: 332, 391, 429, 702, 747, 950, 1099, 1406



This can be effected in the presence of oxygen, light, and a mercury salt. Trimethylamine is an efficient catalyst,²⁶⁵ active even at -40° .⁵³³ The addition goes smoothly in the presence of cuprous mercaptide.¹⁰⁹⁸ The addition of benzyl mercaptan is effected at -20° .^{1416a}

The aldehyde, $\text{PhCH}_2\text{SCMe}_2\text{CHO}$, is an intermediate in the synthesis of penicillamine^{623, 1364, 1374} and has been treated in Chapter 5 of Volume I.

The ethyl sulfide-acetal, $\text{EtSCH}_2\text{CH}_2\text{CH}(\text{OEt})_2$, and the α -butylmercaptopropionaldehyde are from the appropriate halogen compounds and the mercaptides. Ethylmercaptopropionaldehyde results from the addition of ethyl mercaptan to acrolein:

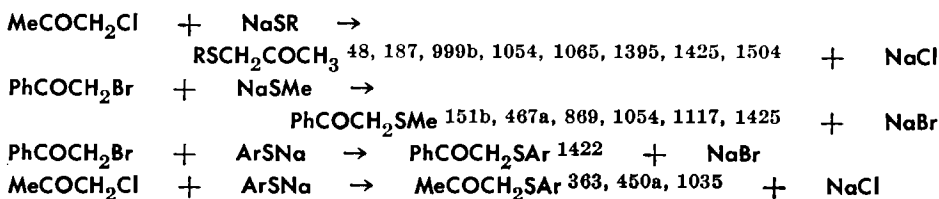


Further action of the mercaptan gives the mercaptal, $\text{EtSCH}_2\text{CH}_2\text{CH}(\text{SEt})_2$.^{1211d, 1211e} Dimethylchloroacetal and sodium sulfide give the sulfide, $[(\text{MeO})_2\text{CHCH}_2]_2\text{S}$.^{1075.5} Crotonaldehyde can be made to take up hydrogen sulfide to form β, β' -thiodibutyraldehyde.⁵⁴³ It combines with ethyl mercaptan in the presence of triethylamine.⁵⁹⁸ Methyl and benzyl mercaptans also have been added to it.²¹⁰

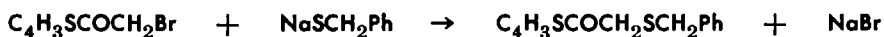
An aromatic aldehydo-sulfide can be made by introducing the $-\text{CHO}$ group into an aromatic sulfide.^{71, 237, 677, 814, 829} The color of the phenylhydrazone of *m*-nitrobenzaldehyde is deepened by the introduction of the $\text{MeS}-$ group in the ortho position and still more in the para.^{656d}

Keto-Sulfides

Haloketones must be brought together gradually with sodium mercaptides with efficient cooling, on account of their great reactivity:

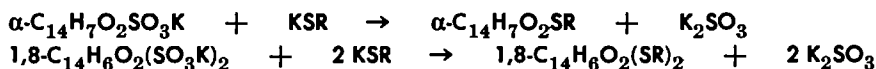


Thenacyl bromide and a mercaptide give a ketosulfide: ^{778a}



Phenacyl and allyl bromides react with RSMgBr to give sulfides. ⁵⁶⁰

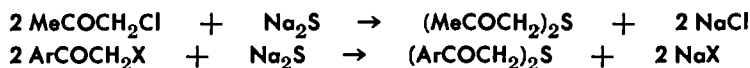
Potassium α -anthraquinonesulfonate reacts with a mercaptide: ^{439, 662, 1159}



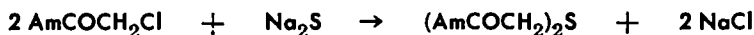
α -Nitroanthraquinone reacts similarly. ^{532b} The monoalkyl anthraquinone thioethers are yellow to orange while the 1,8-dithioethers are deep red. With anthraquinone-1,5-disulfonate, the reaction stops with the replacement of one sulfonic acid group. ^{253, 421, 439, 662, 1159} A number of the α -anthraquinone alkyl sulfides have been given in Chapter 2 of Volume I, under identification of mercaptans. As a sulfonic acid group in the β -position is not split off readily, the β -anthraquinone sulfides have to be made by alkylating β -mercaptoanthraquinone. ^{499, 532b}

A mercaptohydroquinone is alkylated and the product oxidised to the sulfide-quinone. ¹⁵ A chlorinated quinone may react with a mercaptide. ⁵⁸⁷

Symmetrical diketosulfides result from the reaction of chloroacetone ^{155, 678, 929, 1250b} or a phenacyl ^{103, 288, 503, 588, 1250b, 1352} halide on sodium sulfide:



A chloromethyl alkylketone reacts similarly: ⁷¹⁷

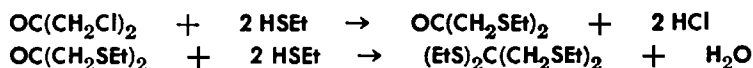


A Mannich base methiodide may be substituted for a halide: ⁵⁵²



The reaction with mercaptides is similar. A bis(chloromethyl) ketone, such as $\text{ClCH}_2\text{CO}(\text{CH}_2)_7\text{COCH}_2\text{Cl}$, may give a polymeric sulfide. ⁵³ Phenacyl sulfide is formed also in the decomposition of a sulfonium compound containing the phenacyl group, such as $\text{Me}_2(\text{PhCOCH}_2)\text{SBr}$. ⁸¹³ A chloromethyl alkyl ketone reacts similarly. ⁷¹⁷

s-Dichloroacetone reacts with two molecules of a mercaptan and then with two more: ^{505, 1193}



A mercaptal, $(\text{RS})_2\text{CHCOCH}_3$, is from dichloroacetone and a mercaptan.^{999a} Aromatic ketosulfides have been made from *p,p'*-dibromobenzophenone and a mercaptide.³⁷⁹ Dimercaptoacetone, $\text{OC}(\text{CH}_2\text{SH})_2$, has been alkylated.^{1250a, 1250c, 1351.5} This can be effected with diazomethane.^{1250c}

Acetylacetone and sulfur dichloride react vigorously. The product appears to be $\text{S}[\text{CH}(\text{COCH}_3)_2]_2$, m. 67 to 72°.²⁸ 3,3'-Diacetyl-4,4'-dihydroxy- α -naphthyl sulfide has been obtained from 2-acetyl- α -naphthol and sulfur chloride.⁹ Sulfenyl chlorides react with ketones.^{1502, 1512} Methanesulfenyl chloride and cyclohexanone give the 1,3-bis-methylmercapto derivative.^{202.5}

From an acid chloride, PhCH_2COCl , and methyl phenyl sulfide, in the presence of aluminum chloride, two products are obtained, $\text{PhCH}_2\text{COC}_6\text{H}_4\text{SMe}$, and $\text{PhCH:C}(\text{C}_6\text{H}_4\text{SMe})_2$, m. 112°.¹⁴⁸⁸ A number of keto-sulfides have been made from aromatic sulfides by the Friedel-Crafts reaction.^{49.5, 54, 74, 194, 236, 243, 322, 380, 381, 680, 815, 816, 1245, 1350}

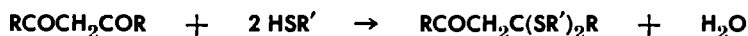
A mercaptan can be added to an unsaturated ketone: ^{450a, 644, 946a, 1018b, 1143, 1380}



Mercaptans, dithiols, and hydrogen sulfide can be added in the presence of piperidine. The products are antioxidants.^{1380, 1383} Hydrogen sulfide is taken up by the unsaturated ketone, $\text{Me}_2\text{C:CHCOCH:CH}_2$, dissolved in alcohol in the presence of sodium acetate. The product is 2,2-dimethyltetrahydro-1,4-thiapyrone, m. 29°, b_{11} 85°.¹⁰²⁹ A polymer is formed by the addition of a dithiol to a doubly unsaturated ketone, such as $(\text{PhCH:CH})_2\text{-CO}$.^{924, 925}

Mesityl oxide takes up one molecule of a mercaptan ^{35, 56, 1114b, 1325} and phorone two.^{1114a} The products are: $\text{Me}_2\text{C}(\text{SR})\text{CH}_2\text{-COMe}$ and $\text{Me}_2\text{C}(\text{SR})\text{CH}_2\text{COCH}_2\text{CMe}_2\text{SR}$. Mesityl oxide combines with hydrogen sulfide to give $\text{S}(\text{CMe}_2\text{CH}_2\text{COMe})_2$.³⁵

Sometimes only one of the carbonyls of a β -diketone reacts with a mercaptan: ¹²¹⁹



The loss of a mercaptan from this leaves an unsaturated keto-sulfide, $\text{RCOCH}:\text{CRSR}'$. This is the case with 1,3-diketocyclohexanone ⁷⁶² and with its 5,5-dimethyl derivative. ²⁴⁹ A dye intermediate may be made in this way. ⁷⁶²

A diazoketo-sulfide is obtained by the reaction of diazomethane on the chloride of a sulfide acid: ⁵¹⁶



A curious reaction takes place when acetophenone, carbon disulfide, and potassium hydroxide are brought together. The product is $\text{PhCOCH}:\text{C}(\text{SK})_2$, from which $\text{PhCOCH}:\text{C}(\text{SH})_2$ can be liberated. This is tautomeric with the dithioacid, $\text{PhCOCH}_2\text{-CSSH}$. It can be alkylated to $\text{PhCOCH}:\text{C}(\text{SR})_2$. ^{758, 759}

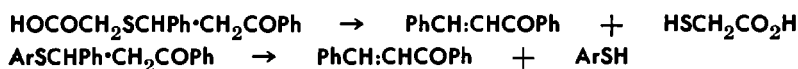
Sulfide ketones have two sets of reactions, those of sulfides and those of ketones. As ketones, they can be reduced by zinc and acid ^{74, 1018a, 1389} or by aluminum isopropoxide ^{54, 236, 1117} to carbinols. Raney nickel may remove the $-\text{SR}$ group and put hydrogen in its place. ¹³⁷⁰ The Wolff-Kishner reduction changes the carbonyl to methylene and may, or may not, affect the sulfide group, according to its location. $p\text{-MeSC}_6\text{H}_4\text{COMe}$ is reduced quantitatively to $p\text{-MeSC}_6\text{H}_4\text{Et}$. ²³⁸

Some keto-sulfides form oximes which may be either *syn* or *anti* in configuration. ^{49.5, 1415} The oximes, $p\text{-MeC}(:\text{NOH})\text{C}_6\text{H}_4\text{-SEt}$ and $p\text{-PhC}(:\text{NOH})\text{C}_6\text{H}_4\text{SEt}$, from the acetyl and benzoyl sulfides, rearrange into the amides. ^{49.5}

With α -keto-sulfides cleavage by alkali may take different directions:



The mercaptan is split off from a β -keto-sulfide: ^{1038a}



A mercaptole from a β -keto-acid loses one molecule of mercaptan: ^{1114a, 1114b}



These may be considered as reversals of the addition of mercaptans to unsaturated ketones.

The normal reactions take place with Grignard reagents.^{49.5, 1018a, 1351.5}

Sulfonium salts are formed with alkyl halides.^{814, 1222}

Certain keto-sulfides are claimed as polymerization modifiers,¹³⁸⁴ others are recommended as stabilizers for fats and oils.^{282, 1381, 1382} The methylmercapto hemi-ketole of methylalloxan and the methylmercapto hemi-acetal of pentaacetyl-aldehyd-galactose are said to aid in developing flavor.^{807.5} Dyes have been made from 1-amino-2,4-bis(β -hydroxyethylmercapto)anthraquinone.²⁶⁷

Cyano-Sulfides

Cyano-sulfides, or sulfide-nitriles, are of interest on account of their close connection with sulfide-acids which will be included in Volume III.

The reaction of ammonium, or potassium, sulfide with chloroacetonitrile gives bis-(cyanomethyl) sulfide, $S(CH_2CN)_2$.^{1322, 1323, 1522}

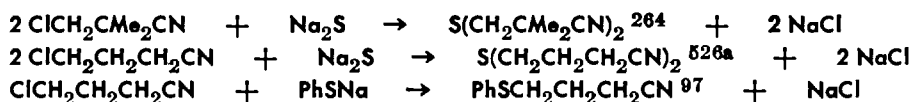
Most of the compounds in this group have the $—CN$ group in the β -position to the sulfur since they are obtained readily by the addition of hydrogen sulfide, or mercaptans, to acrylonitrile. This subject has been reviewed by Bayer.⁹³ Acrylonitrile and hydrogen sulfide unite, in the presence of 0.3% of trimethyl benzylammonium hydroxide, or of sodium methylate, to form the sulfide, $S(CH_2CH_2CN)_2$. Without a catalyst, there is no reaction.^{543, 695} The same compound is obtained by contacting acrylonitrile with sodium trithiocarbonate,⁶⁶⁶ or with viscose.⁸⁹¹ The product has been suggested for use in the solvent extraction of hydrocarbons.^{20b, 944}

Cyano-sulfides, $RSCMe_2CN$, have been prepared by the reaction of $Me_2C(CN)$ radicals on mercaptans.²¹⁴

The addition of mercaptans to acrylonitrile has been discussed in Chapter 1 under the formation of sulfides by addition. Reference should be made to this. Thiophenol has been added to β -alkylated acrylonitrile¹²⁰² and to 1-cyanocyclohexene.¹²⁰⁴ A sulfide-alcohol, $ArCH_2CH_2SCH_2CH_2OH$, has been added to acrylonitrile to make an ether-sulfide nitrile.³⁵⁴ A study has been made of the addition of mercaptans to α -substituted acrylonitrile.

Piperidine is an efficient catalyst. *t*-Butyl mercaptan gave low yields.¹²⁰³

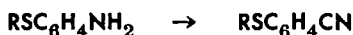
Halonitriles react regularly with alkali sulfide or mercaptides:



A halosulfide and sodium cyanide give a sulfide-nitrile:¹¹



By the diazo reaction, an alkyl-mercaptoaniline can be converted to a nitrile: ^{240, 829, 1510a}



γ -Chlorobutyronitrile reacts regularly with an alkali sulfide or mercaptide. Two of the products that have been made from it are $\text{S}(\text{CH}_2\text{CH}_2\text{CH}_2\text{CN})_2^{526a}$ and $\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{CN}^{97}$

The reactions of the sulfide-nitriles are those characteristic of sulfides and nitriles. Many sulfide acids have been made by hydrolyzing them. The presence of a $-\text{CN}$ or $-\text{CO}_2\text{H}$ group in the β -position labilizes the sulfide linkage. With 10% aqueous sodium hydroxide, 93% of the calculated tolyl mercaptide, *p*- $\text{MeC}_6\text{H}_4\text{SNa}$, was obtained from the sulfide, $\text{MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{CN}^{886}$

Ortho esters, $\text{MeSCH}_2\text{CH}_2\text{C}(\text{OMe})_3$ and $\text{MeSCH}_2\text{CH}_2\text{C}(\text{OEt})_3$, have been prepared from β -methylmercaptopropionitrile.²⁹⁴

CHAPTER 7.

Physical Properties of Some Substituted Sulfides

This chapter lists a large number of substituted sulfides, arranged in series according to their structures. No claim is made for completeness. Available data on the properties are given for each compound, with references. In this way the state of our knowledge of each compound is shown and the reader is directed to the sources from which further information may be obtained.

α - and β -Hydroxy-Sulfides

MeSCH₂OAc, b_{11} 53–5°, b_{20} 60–2°. ^{780a}

EtSCH₂OH, d 14/4 1.070. ^{848b}

PrSCH₂OH, d 14/4 1.018. ^{848b}

i-AmSCH₂OH, d 14/4 0.924. ^{848b}

PhSCH₂OH, d 14/4 1.182; ^{848b} Ac., b_{713} 249°, b_{16} 139–46°. ¹¹³⁵

PhCH₂SCH₂OH, $b_{0.1}$ 84–6°. ^{150.5}

MeSCH₂CH₂OH, b_{15} 58–68°, ¹⁴⁶⁸ b_{16} 68–70°, ^{780a} b_{20} 68–70°, ¹³³⁸, ¹⁴⁶⁹ b_{29} 78–9°, ^{674.5} b_{30} 80.5–1°, b_6 72°; d 20/4 1.0622; n 30/D 1.4867; ^{780a} Ac., b_{15} 68–70°, ²⁷² b_{21} 79–80°; d 30/4 1.0500; n 30/D 1.4583. ^{674.5}

EtSCH₂CH₂OH, b . 184°, ³⁶⁷ 182–4°, ¹³²³ 181.5–2.0° (cor.), ^{989d} b_{28} 99°, ¹²⁷ b_{14} 80°. ¹²⁵¹

- $\text{BuSCH}_2\text{CH}_2\text{OH}$, b_2 $82-4^\circ$,⁸⁸⁶ b_3 $92-3^\circ$,^{1455a} b_{23} 118° ; ¹²⁷ d $0/4$ 0.9826 , d $25/4$ 0.9665 ; n $20/D$ 1.4800 ; Ac. , b_4 84° ; d $0/4$ 1.0041 , d $25/4$ 0.9846 ; n $20/D$ 1.4648 .^{1455a}
 $s\text{-BuSCH}_2\text{CH}_2\text{OH}$, b_{42} $105-8^\circ$.⁶⁸⁹
 $t\text{-BuSCH}_2\text{CH}_2\text{OH}$, b_{45} $111-4^\circ$,⁶⁸⁹ b_4 $60-4^\circ$, b_{13} $96-8^\circ$,¹³⁰⁴ b_{17} $89-93^\circ$.^{350d}
 $i\text{-AmSCH}_2\text{CH}_2\text{OH}$, b_{10} $110-1^\circ$; d_{16} 0.948 ; n_D 1.475 .¹²⁷⁷
 $t\text{-AmSCH}_2\text{CH}_2\text{OH}$, b_{8-10} $100-2^\circ$.¹³⁰⁴
 $\text{HepSCH}_2\text{CH}_2\text{OH}$, b_{33} $153-4^\circ$.⁵⁷⁵
 $\text{OctSCH}_2\text{CH}_2\text{OH}$, b_{12} $146-51^\circ$.⁵⁸¹
 $\text{NonSCH}_2\text{CH}_2\text{OH}$, b_{20} $174-6^\circ$.⁵⁷⁵
 $\text{C}_{11}\text{H}_{23}\text{SCH}_2\text{CH}_2\text{OH}$, m . 27° .⁵⁷⁵
 $\text{C}_{12}\text{H}_{25}\text{SCH}_2\text{CH}_2\text{OH}$, m . 28.5° ; $b_{1,2}$ 145° .¹³²⁵
 $\text{C}_6\text{H}_{11}\text{SCH}_2\text{CH}_2\text{OH}$, b_5 $115-25^\circ$.⁵⁸¹
 $\text{CH}_2:\text{CHCH}_2\text{SCH}_2\text{CH}_2\text{OH}$, b_{12} $90-2^\circ$; d $15/4$ 1.0316 ; Ac. , b_{12} 95° ; d $20/4$ 1.0451 .¹²⁷⁶
 $\text{PhSCH}_2\text{CH}_2\text{OH}$, b_4 $119-20^\circ$,^{1034b} b_2 $115-6^\circ$,^{781b} b_7 $134-5^\circ$,^{21a} b_8 135° ,⁴⁸⁰ b_9 $136-7^\circ$; ¹²⁶⁰ d $20/4$ 1.1431 ,^{781b} d $24/4$ 1.1136 ; ⁴⁸⁰ n $20/D$ 1.5917 ,^{781b} n $22/D$ 1.5870 ,⁴⁸⁰ n $25/D$ 1.5897 ,^{21a} 1.5892 ; ¹²⁶⁰ Ac. , b_8 $106-8^\circ$; Bz. , b_{10} 180° ; ^{442.5} $p\text{-nitrobenzoate}$, m . 59° ; $2,4\text{-dinitrobenzoate}$, m . 113° .⁷⁷²
 $p\text{-MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{OH}$, b . $282-3^\circ$, b_{30} 174° ,⁵⁰⁶ b_1 $119-20^\circ$.⁸⁸⁶
 $2,4,6\text{-Me}_3\text{C}_6\text{H}_2\text{SCH}_2\text{CH}_2\text{OH}$, m . 25° .^{66a}
 $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{OH}$, b_{18} 169° ,⁵⁰⁴ b_1 70.5° ; n $26/D$ 1.5542 .¹³⁴⁷
 $\text{Ph}_3\text{CSCH}_2\text{CH}_2\text{OH}$, m . 115° .^{331b}
 $\beta\text{-C}_{10}\text{H}_7\text{SCH}_2\text{CH}_2\text{OH}$, m . 64° .^{66a}
 $3\text{-C}_4\text{H}_9\text{SCH}_2\text{CH}_2\text{OH}$, b_1 125° ,²⁰⁹ b_2 $116-7^\circ$.⁶⁷⁵
 $\text{EtSCHMeCH}_2\text{OH}$, b_{17} $80-90^\circ$; n $20/D$ 1.4777 .⁵²³
 $3\text{-C}_4\text{H}_9\text{SCHMeCH}_2\text{OH}$, b_4 111° .⁶⁷⁵
 $\text{MeSCH}_2\text{CHMeOH}$, b_7 $48-51^\circ$,^{166a} b_{20} $69-70^\circ$; ^{924.5} 67° ; d $20/4$ 1.037 ; n $20/D$ 1.4869 ,⁶⁸⁴ 1.4817 .^{924.5}
 $\text{EtSCH}_2\text{CHMeOH}$, b_7 $60-1^\circ$,^{166a} b_{15} 76.5° ; n $20/D$ 1.4734 .⁵²³
 $t\text{-BuSCH}_2\text{CHMeOH}$, b_{10} $88-92^\circ$.¹³⁰⁴
 $t\text{-AmSCH}_2\text{CHMeOH}$, b_{8-10} $98-104^\circ$.¹³⁰⁴
 $\text{PhSCH}_2\text{CHMeOH}$, b_2 $110-2^\circ$,¹²⁵⁸ b_9 133° ; ¹²⁶⁰ d_{25} 1.1014 ; n $20/D$ 1.5715 ,¹²⁵⁸ n $25/D$ 1.5715 ; $p\text{-nitrobenzoate}$, m . 65° ,¹²⁶⁰ 64° ; $2,4\text{-dinitrobenzoate}$, m . 110° ; ⁷⁷² $3,5\text{-dinitrobenzoate}$, m . 113.5° .¹²⁵⁸
 $\text{PhCH}_2\text{SCH}_2\text{CHMeOH}$, b_{11} $55-6^\circ$.^{1211a}
 $\text{MeSCH}_2\text{CHPrOH}$, b_{18} 90° ; d $20/4$ 0.941 ; n $20/D$ 1.4792 .⁵⁶⁹
 $\text{PrSCH}_2\text{CHHexOH}$, $b_{0.03}$ $67-70^\circ$, $b_{0.25}$ $92-3^\circ$.⁷⁶⁴

$C_{12}H_{25}SCH_2CH(CH:CH_2)OH$, $b_{0.2}$ 135–7°; d 20/4 0.9050; n 20/D 1.4802.⁹²⁶

$C_{16}H_{33}SCH_2CH(CH:CH_2)OH$, m . 27.5°; $b_{0.15}$ 163–4°.⁹²⁶

$MeSCH_2CHPhOH$, b_{12} 141–2°,¹¹¹⁷ $b_{0.8}$ 127°; ^{513, 558} d 20/4 1.1062; n 20/D 1.5690.⁵⁵⁸

$EtSCH_2CHPhOH$, $b_{0.1}$ 96–101°.¹¹¹⁷

$PrSCH_2CHPhOH$, b_1 117–8°.⁷⁶⁴

$C_{12}H_{25}SCH_2CHPhOH$, $b_{0.2}$ 178–80°; d 20/4 0.9044; n 20/D 1.5130.⁹²⁸

$C_{16}H_{33}SCH_2CHPhOH$, $b_{0.3}$ 220–1°; d 20/4 0.9413; n 20/D 1.5070.⁹²⁸

$MeSCH_2CH(C_6H_4Me-p)OH$, $b_{0.2}$ 94–5°.¹¹¹⁷

$EtSCH_2CH(C_6H_4Me-p)OH$, $b_{0.2}$ 98–9°.¹¹¹⁷

$MeSCHMeCHPhOH$, $b_{0.8}$ 85–7°.¹¹¹⁷

$EtSCHMeCHPhOH$, $b_{0.1}$ 86–8°.¹¹¹⁷

$MeSCH_2CMePhOH$, b_{12} 138–9°.¹¹¹⁷

$EtSCH_2CMePhOH$, b_{12} 130–50°.¹¹¹⁷

$EtSCH_2CPh_2OH$, m . 41.5°.¹¹¹⁷

Other Hydroxy-Sulfides

$MeSCH_2CH_2CH_2OH$, b_{24} 102°,^{111c} b_{30} 105–5.5°; d 20/4 1.0296,^{780a} 1.030;^{111c} n 30/D 1.4832.^{780a}

$EtSCH_2CH_2CH_2OH$, b_{15} 104°.^{1211a}

$PrSCH_2CH_2CH_2OH$, b_{18} 115°,¹⁰⁸⁵ 112°,¹⁰⁹ b_{23} 118°; ¹²⁶⁰ d 20/4 0.9794; ¹⁰⁹ n 25/D 1.4794.¹²⁶⁰

$BuSCH_2CH_2CH_2OH$, $b_{0.8}$ 80°; n 25/D 1.4782.¹²⁶⁰

$AmSCH_2CH_2CH_2OH$, b_{10} 120–5°.⁸³

$PhSCH_2CH_2CH_2OH$, b_{19} 168°,²⁴⁴ b_{20} 185–8°,¹¹⁹³ b_8 155–9°,¹²⁶⁰ b_2 134–5°; ^{781b, 1260} d 20/4 1.1178; ^{781b} n 26/D 1.5646,²⁴⁴ n 20/D 1.5813,^{781b} n 25/D 1.5792; ¹²⁶⁰ p -nitrobenzoate, m . 60°; 2,4-dinitrobenzoate, m . 85°.⁷⁷²

$MeC_6H_4SCH_2CH_2CH_2OH$, b_2 145–6°.⁸⁸⁶

$PhCH_2SCH_2CH_2CH_2OH$, b_1 119.5°,¹³⁴⁷ b_{19} 185°,^{1211a} b_{22} 185°,¹²⁶⁰ b_{20} 185–8°; ¹¹⁹³ n 21/D 1.5711,¹³⁴⁷ n 25/D 1.5632.¹²⁶⁰

$EtS(CH_2)_4OH$, b_{19} 120°; d 20/4 0.9794; n 20/D 1.48118.¹⁰⁹

$BuS(CH_2)_4OH$, b_8 129–30°.⁶⁸⁹

$PhS(CH_2)_4OH$, m . 24°; ¹⁰⁹ p -nitrobenzoate, m . 60°; 2,4-dinitrobenzoate, m . 86°.⁷⁷²

p - $MeC_6H_4S(CH_2)_4OH$, b_2 161°.⁸⁸⁶

3- $C_4H_9S(CH_2)_4OH$, $b_{1.5}$ 134–5°.⁶⁷⁵

- $\text{MeSCH}_2\text{CH}_2\text{CHPhCPh}_2\text{OH}$, m. 111° .⁷⁴⁵
 $\text{MeS}(\text{CH}_2)_5\text{OH}$, b_{16} 121° ; d 20/4 0.9846; n 20/ α 1.488185.¹⁰⁹
 $\text{EtS}(\text{CH}_2)_5\text{OH}$, b_{20} 135° .¹¹³
 $\text{PhS}(\text{CH}_2)_5\text{OH}$, m. 31.5° ; ¹⁰⁹ *p*-nitrobenzoate, m. 55° ; 2,4-dinitrobenzoate, m. 74° .⁷⁷²
 $\text{EtS}(\text{CH}_2)_6\text{OH}$, b_{17} $134-6$.¹¹³
 $\text{PhS}(\text{CH}_2)_6\text{OH}$, m. 43° ; ^{112b} *p*-nitrobenzoate, m. 109° ; 2,4-dinitrobenzoate, m. 140° .⁷⁷²
 $\text{MeS}(\text{CH}_2)_7\text{OH}$, b_{10} $133-4^\circ$.¹⁰⁷
 $\text{PhS}(\text{CH}_2)_7\text{OH}$, m. 49° .^{112b}
 $\text{MeS}(\text{CH}_2)_8\text{OH}$, m. 12° ; b_{10} $135-8^\circ$.¹⁰⁷
 $\text{PhS}(\text{CH}_2)_8\text{OH}$, m. 55° .^{112b}
 $\text{MeS}(\text{CH}_2)_9\text{OH}$, m. 22° ; b_9 $138-42^\circ$.¹⁰⁷
 $\text{PhS}(\text{CH}_2)_9\text{OH}$, m. 60° .^{112b}
 $\text{MeS}(\text{CH}_2)_{10}\text{OH}$, m. 25° ; b_{13} $170-2^\circ$.¹⁰⁷
 $\text{PhS}(\text{CH}_2)_{10}\text{OH}$, m. 66.5° .^{112b}
 $\text{MeS}(\text{CH}_2)_{12}\text{OH}$, m. 49° .¹⁰⁷
 $\text{MeS}(\text{CH}_2)_{14}\text{OH}$, m. 38° .¹⁰⁷
 $\text{MeS}(\text{CH}_2)_{16}\text{OH}$, m. 56° .¹⁰⁷
 $\text{MeS}(\text{CH}_2)_{18}\text{OH}$, m. 62° .¹⁰⁷

Cyclic Hydroxy-Sulfides

- 2-MeSC₆H₁₀OH, b_{11} $97-8^\circ$.¹⁵³
2-C₅H₉SC₅H₈OH, b_{12} 157° ; d_{25} 1.069; n 25/D 1.5240.^{1005b, 1006}
2-C₅H₉SC₆H₁₀OH, b_{12} 165° ; d_{25} 1.067; n 25/D 1.5250.^{1005b, 1006}
2-C₆H₁₁SC₆H₁₀OH, b_{12} 170° ; d_{25} 1.051; n 25/D 1.5231.^{1005b}
2-PhSC₆H₁₀OH, b_{22} 207° ; d_{25} 1.137; n 25/D 1.5825.¹⁰⁰⁶
o-MeSC₆H₄OH, b_{22} 105° ; d 25/4 1.165.⁶⁷⁰
m-MeSC₆H₄OH, m. 15° ; b. 224° , b_{14} $148-51^\circ$; ^{1506a} pK 9.53.^{168b}
p-MeSC₆H₄OH, m. 85° ; ^{1506b} b_6 113° ; ^{976b} Ac., m. 44° ; ^{1506b} pK 9.53.^{168b}
p-EtSC₆H₄OH, m. 41° ; ^{846, 976b} b. $282-7^\circ$, ⁸⁴⁶ b_{13} $153-5^\circ$.^{976b}
p-PrSC₆H₄OH, m. 33.5° , ¹³⁴⁰ 37° ; b_{5-8} $150-3^\circ$.^{976b}
i-PrSC₆H₄OH-*p*, b_{13} $150-2^\circ$.^{976b}
o-BuSC₆H₄OH, b_5 $110-2^\circ$, ^{976b} b_{10} 125° ; d 25/4 1.061.⁶⁷⁰
m-BuSC₆H₄OH, b_4 $127-40^\circ$.^{976b}
p-BuSC₆H₄OH, m. 50° , ^{976b} 37° ; ¹³⁴⁰ b_7 $166-8^\circ$.^{976b}
i-BuSC₆H₄OH-*p*, b_{10} $159-62^\circ$.^{976b}
p-AmSC₆H₄OH, m. 62.5° , ⁹⁷⁵ 56° , ¹³⁴⁰ 59° ; b_{3-4} 164° ; b_{8-10} $175-80^\circ$.^{976b}

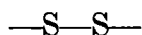
- i*-AmSC₆H₄OH-*p*, *b*₃ 150–2°. ^{978b}
p-HexSC₆H₄OH, *m*. 59°, ¹³⁴⁰ 60°; *b*₅ 178–80°, *b*₁₈ 194–6°. ^{978b}
o-PhSC₆H₄OH, *m*. 44°; ⁶⁷⁰ *b*₁ 136–40°, ¹¹²⁹ *b*₃ 140°, ⁶⁴⁸ *b*₆₆ 219°. ¹⁷⁹
m-PhSC₆H₄OH, *b*₃ 159–61°. ⁶⁴⁸
p-PhSC₆H₄OH, *m*. 51°, ⁶⁴⁸ 25°; ¹⁷⁹ *b*₃ 164–5°, ⁶⁴⁸ *b*_{1.5} 178–80°. ¹¹²⁹
p-PhCH₂SC₆H₄OH, *m*. 104°; *b*₁₅ 220–5°. ^{978b}
p-MeC₆H₄SC₆H₄OH-*p*, *m*. 68°, ⁶⁴⁸ 164°. ^{443a}
2,4-MeS(Me)C₆H₃OH, *b*₁₈ 118–20°. ¹⁵⁰¹
2,4-PhCH₂S(Me)C₆H₃OH, *b*₂₁ 190–2°; Ac., *m*. 63°. ¹⁵⁰¹
4,2-MeS(Me)C₆H₃CH₂OH, *b*_{16.5} 162–2.5°. ⁸¹⁴
p-MeSC₆H₄CHMeOH, *m*. 40°. ⁵⁴
p-MeSC₆H₄CHEtOH, *b*₁₇ 160–2°. ²³⁶
p-MeSC₆H₄CHPhOH, *m*. 99°, ^{1018a} 93°; ⁷⁴ Ac., *b*₁₅ 221°; *d* 21/4
1.144; *n* 20/D 1.5912. ⁷⁴
MeSC₆H₄CPh₂OH, *ortho*, *m*. 96°; ^{192a} *meta*, *d* 20/4 1.1409; *n* 90/4
1.62067; ¹⁸⁸ *para*, *m*. 67°. ¹⁹⁴
o-EtSC₆H₄CPh₂OH, *m*. 94°. ¹⁹³
p-MeSC₆H₄CPh(CH₂Ph)OH, *m*. 117°. ^{1018a}
1-MeSC₁₀H₆OH-4, *m*. 110°; ¹⁵¹⁷ Ac., *m*. 65°. ¹⁵¹⁷
1-MeSC₁₀H₆OH-5, *m*. 155°. ¹¹⁶⁴
2-MeSC₁₀H₆OH-6, *m*. 121°; Ac., *m*. 74°. ¹⁵⁰⁵
1-PhCH₂SC₁₀H₆OH-4, *m*. 105°. ¹⁵¹⁷
1-PhCH₂SC₁₀H₆OH-5, *m*. 130°; Ac., *m*. 157°. ¹¹⁶⁴
1-(*p*-MeC₆H₄S)C₁₀H₆OH-2, *m*. 84°. ²⁰⁸
1-(β-C₁₀H₇S)C₁₀H₆OH-2, *m*. 92°. ²⁰⁸

Symmetrical Cyclic Hydroxy-Sulfides

- (*o*-HOC₆H₄)₂S, *m*. 142°; Ac., *m*. 96°. ^{932a}
(*m*-HOC₆H₄)₂S, *m*. 130°, ^{1363b} 129°; ⁶²², ^{1362b}, ^{1363a} Ac., *m*. 87°. ^{1363b}
(*p*-HOC₆H₄)₂S, *m*. 152°, ⁹⁴³ 151.5°, ⁸⁰⁵ 151°, ⁴⁰⁶ 150°, ⁸⁷⁶, ^{1362a}, ^{1363a}
149°, ⁸⁷⁵ 144°; ^{803b} Ac., *m*. 94°, ^{1362a}, ^{1363a} 93°. ⁸⁰⁵
(2-HOC₆H₃Me-5)₂S, *m*. 143°, ¹⁵⁸, ⁵³⁸ 135°. ¹³⁹⁹
(4-HOC₆H₃Me-2)₂S, *m*. 143.5°. ⁴⁰⁶
(4-HOC₆H₃Me-3)₂S, *m*. 124°; Ac., *m*. 44°. ^{1363b}
(4-HOC₆H₂Me₂-2,5)₂S, *m*. 189°. ⁹⁴³
(2-HOC₆H₂Me₂-4,5)₂S, *m*. 163°. ⁹⁴³
(2-HOC₆H₂Me₂-3,5)₂S, *m*. 98°. ⁸⁴⁵
(2-HOC₆HMe₃-3,5,6)₂S, *m*. 128°. ⁸⁴⁵
(4-HOC₆H₃MePr-*i*-2,5)₂S, *m*. 153.5°, ⁴⁰⁶ 153°. ^{1362b}, ^{1363a}
(2-HOC₆H₂MeBu-*t*-4,5)₂S(?), *m*. 158°. ¹²⁸⁶

- (2,4-(HO)₂C₆H₃)₂S, m. 187°,¹⁰⁹⁴ 167°.⁴⁰⁶
 (2-HOCH₂C₆H₄)₂S, m. 164°.¹¹⁶¹
 2-Hydroxycyclopentyl, m. 45°,¹⁰⁰⁶ 44°; ^{1005a} b₂₀ 205°; ^{1005a}, ¹⁰⁰⁶
 n 25/D 1.5370.¹⁰⁰⁶
 2-Hydroxycyclohexyl, stereoisomers, m. 71°,^{1005b} 72°,^{1005a}, ¹⁰⁰⁶ 90°
 and 104°; ¹⁰⁰⁶ b₂₀ 215°; ^{1005a}, ¹⁰⁰⁶ diAc., m. 62°.^{1005a}
 2-Hydroxycycloheptyl, m. 88°; ^{1005a}, ¹⁰⁰⁶ b₂₀ 225°,^{1005a} 230°.¹⁰⁰⁶
 2-Hydroxymethylcyclohexyl, m. 55°.^{1005b}
 3-Hydroxy-2,3-dihydro-2-indenyl, m. 136°.¹⁰⁰⁶
 3-Hydroxy-1,2,3,4-tetrahydronaphthyl-2, m. 151°.^{1005a}, ¹⁰⁰⁶
 β-Hydroxy-α-naphthyl, 2 forms, m. 153°,^{1042a} 152°; m. 216°,^{649b}
 215°,¹⁰⁶⁰, ^{1362b}, ^{1363a} 214°,^{335c}, ¹⁴⁰³ 226°,¹¹⁸³ 212°,⁹, ²⁸⁷ 211°; ⁶³²,
 ^{1042a} Ac., 200°; ^{1363b} 193°; ⁶³² 154°; ¹⁰⁶⁰ Bz., m. 208°.¹⁰⁶⁰
p-HOC₆H₄CH(SMe)CH(SMe)C₆H₄OH-*p*, m. 195°; diAc., m.
 119°.¹⁰⁵⁷
 1,4-(HO)₂C₆(SEt)₄-2,3,5,6, m. 58°.⁵⁸⁷

Hydroxy Multiple Sulfides

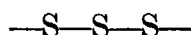


- MeSCH₂CH(SMe)CH₂OAc, b₁₂ 124°.⁸⁰⁰
 EtSCH₂CH(SEt)CH₂OAc, b₁₂ 145°.⁸⁰⁰
 MeSCH₂CH₂SCH₂CH₂OH, b₂ 125°.²¹¹
 EtSCH₂CH₂SCH₂CH₂OH, b. 278°.³⁶⁷
 PrSCH₂CH₂SCH₂CH₂OH, b₁₀ 151–4°.⁵⁷⁵
 BuSCH₂CH₂SCH₂CH₂OH, b₁₀ 163–5°.⁵⁷⁵
 MeSCH₂CH(OH)CH₂SMe, b₅₀ 161°; d 25/4 1.1256; n 25/D
 1.5359.¹³⁹¹
 EtSCH₂CH(OH)CH₂SEt, b_{1.0} 94°; d 25/4 1.0528; n 25/D
 1.5148.¹³⁹¹
 PrSCH₂CH(OH)CH₂SPr, b_{0.7} 105°; d 25/4 1.0144; n 25/D
 1.5063.¹³⁹¹
 BuSCH₂CH(OH)CH₂SBu, b_{1.0} 133°; d 25/4 0.9867; n 25/D
 1.5007.¹³⁹¹
 AmSCH₂CH(OH)CH₂SAm, b_{0.8} 148°; d 25/4 0.9692; n 25/D
 1.4960.¹³⁹¹
 HexSCH₂CH(OH)CH₂SHex, b_{1.5} 184°; d 25/4 0.9547; n 25/D
 1.4927.¹³⁹¹
 EtSCH₂CH(OH)CH₂SCH₂Ph, b_{0.2} 162–3°.^{1211a}
 (PhCH₂SCH₂)₂CHOH, m. 59°; ⁵⁰⁵ b_{0.2} 215°.^{1211a}
 (*p*-MeSC₆H₄)₂CHOH, m. 98°.^{1018a}

(*o*-MeSC₆H₄)₂CPhOH, m. 136°. ^{192a}

(*o*-EtSC₆H₄)₂CPhOH, m. 124°. ¹⁹³

3,5-(BuSCH₂)₂C₆H₂MeOH-4, n 20/D 1.5490. ¹²¹⁴



(*p*-MeSC₆H₄)₃COH, m. 128°. ^{192b}

(*o*-EtSC₆H₄)₃COH, m. 96°. ¹⁹³

(*o*-PhSC₆H₄)₃COH, m. 180°. ^{192b}



(*p*-MeSC₆H₄)₂C(OH)C(OH)(C₆H₄SMe-*p*)₂, m. 169°. ^{1018a}

Sulfide-Glycols

HOCH₂CH₂SCH₂CH₂OH, thiodiglycol, m. -10°, ²⁹⁵ -16°; ⁴³⁰ b_{0.005} 104°, ⁴³⁰ b_{0.1} 121.8°, ^{989c} ^{989d} b₁ 133.3°, ^{989d} b₂ 130°, ²⁸³ ²⁸⁴ b₆ 147°, ^{1482b} 147.5°, ²⁹⁵ b₁₄ 168°, ³⁵² b₂₀ 165°, ²⁹⁵ 164-6°, ⁴³⁶ b₄₀ 181.5°; ²⁹⁵ vapor pressure 0.7 mm. at 10°, 0.8 mm. at 20°, 0.9 mm. at 30°, 1.1 mm. at 40°, 1.5 mm. at 50°, 1.9 mm. at 60°, 2.3 mm. at 70°; ⁹⁰ d 0/4 1.1973, ²⁹⁵ d 20/4 1.1817, ^{989c} 1.1819, ⁴³⁰ d 25/4 1.1793, ²⁹⁵ 1.1770; ³⁰⁷ n 20/D 1.52031, ⁴³⁰ n 26/D 1.5146, ²⁹⁵ n 25/D 1.5191; ³⁰⁷ parachor, 279.9 at 20°. ^{989c}

HOCH₂CH₂SCH₂CHMeOH, b_{0.06} 94-6°, ³⁵² b₂ 114-5°, ¹⁰⁶⁸ b_{2.5} 120-5°, ⁵⁹⁹ b₈ 154°, ¹⁴⁶² b₃₂ 193-7°; ¹¹⁹² d 25/4 1.126; n 25/D 1.5079, ³⁵² 1.5091; ¹⁰⁶⁸ viscosity at 25° 68.4; ³⁵² *p*-nitrobenzoate, m. 86°, ³⁵² 85°. ⁵⁹⁹

HOCH₂CH₂SCH₂CHMeOAc, b_{0.009} 76-8°; d 25/4 1.124; n 25/D 1.5070; viscosity at 25° 71.8; Ac., b₃ 104°; d 25/4 1.112; n 25/D 1.4792; viscosity at 25° 14.06. ³⁵²

HOCH₂CH₂SCHMeCH₂OH, b_{0.005} 82-3°, ³⁵² b_{0.5} 110-6°, ⁵²³ b₅ 136-7°; ⁵⁹⁹ ¹⁴⁶² d 25/4 1.125; ³⁵² n 20/D 1.5103, ⁵²³ n 25/D 1.5078; viscosity at 25° 66.4; ³⁵² *p*-nitrobenzoate, m. 90°. ³⁵²

S(CH₂CHMeOH)₂, b₄ 125°, ¹⁴⁶² b₂₅ 165-70°. ¹¹⁹²

S(CH₂CMeEtOH)₂, b₁₁ 148.5-9°. ⁹⁰⁵

S(CH₂CPh₂OH)₂, m. 106-8°. ¹¹⁰¹

S[CH₂CPh(α-C₁₀H₇)OH]₂, m. 78-80°. ¹¹⁰¹

HOCH₂CH₂SCH₂CH₂CH₂OH, b₁₂ 164-6°, ⁵⁸¹ b_{2.5} 144°, ⁵⁹⁹ b_{0.07} 110-1°; d 25/4 1.1385; n 25/D 1.5105; viscosity at 25° 63; ³⁵² *p*-nitrobenzoate, m. 110°, ⁵⁹⁹ 108°; ³⁵² *p*-Ph-phenoxyacetate, m. 107°. ³⁵²

HOCHMeCH₂SCH₂CH₂CH₂OH, b₃₂ 193-7°. ¹¹⁹²

$\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_4\text{OH}$, $b_{0.014}$ 112° ; d 25/4 1.106; n 25/D 1.5091; viscosity at 25° 67.1.³⁵²

$\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_5\text{OH}$, $b_{0.009}$ 110° ; d 25/4 1.074; n 25/D 1.4995; viscosity at 25° 57.9.³⁵²

$\text{S}(\text{CH}_2\text{CH}_2\text{CH}_2\text{OH})_2$, bis-Ph urethane, m . 148° ,^{111a} 123° ,⁸⁹⁰ 120° ; ¹²⁶⁰ di-*p*-nitrobenzoate, m . 105° .^{111b}

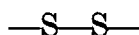
$\text{PhSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{OH}$, m . 67° .⁶⁹⁰

p- $\text{MeC}_6\text{H}_4\text{SCH}_2\text{CH}(\text{OH})\text{CH}_2\text{OH}$, m . 58° ; diAc., $b_{0.3}$ 166° .^{331a}

$\text{C}_{12}\text{H}_{25}\text{SCH}_2(\text{CHOH})_4\text{CH}_2\text{OH}$, m . 107° .^{445, 832}

$\text{S}[\text{CH}_2(\text{SH})\text{CH}(\text{OH})\text{CH}(\text{OH})\text{CH}(\text{OH})\text{CH}(\text{OH})\text{CH}_2\text{OH}]_2$, m . 142° .¹²⁴⁰

9,10-Dihydroxy-9,10-diphenyl-9,10-dihydro- α -anthracenylmethyl sulfide, m . 165° ; diphenyl sulfide, m . 240° ; dibenzyl sulfide, m . 140° .¹⁰⁷⁰



$\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{SCH}_2\text{CH}_2\text{OH}$, m . 18° ; ⁵³⁰ b_5 $52-4^\circ$,¹⁴⁵⁰ $b_{0.005}$ $114-5^\circ$.⁵³⁰

$\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$, m . 64° ; ^{104a}, ¹¹⁴, ⁹⁷⁴, ¹¹⁵² $b_{0.5}$ 170° .⁹⁷⁴

$\text{HOCHMeCH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CHMeOH}$, m . 28° ; b_5 $189-90^\circ$,⁵³⁰ $b_{0.035}$ 128° ; d 25/4 1.130; n 25/D 1.5362; viscosity at 25° 137.4.³⁵²

$\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_3\text{SCH}_2\text{CH}_2\text{OH}$, m . 18° ; b_4 200° .⁵³⁰

$\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_4\text{SCH}_2\text{CH}_2\text{OH}$, m . 35° ,¹¹⁵² 30° ; b_3 207° .⁵³⁰

$\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_5\text{SCH}_2\text{CH}_2\text{OH}$, m . 36° ,¹¹⁵² 30° ; b_3 210° .⁵³⁰

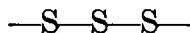
$\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_6\text{SCH}_2\text{CH}_2\text{OH}$, m . 45° ; $b_{0.02}$ 130° .⁵³⁰

$\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_8\text{SCH}_2\text{CH}_2\text{OH}$, m . 61° .⁵³⁰

$\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_9\text{SCH}_2\text{CH}_2\text{OH}$, m . 64° .⁵³⁰

$\text{HOCH}_2\text{CH}_2\text{S}(\text{CH}_2)_{10}\text{SCH}_2\text{CH}_2\text{OH}$, m . 70° .⁵³⁰

$\text{HOCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{OH}$, m . 52° ; ³⁵², ⁷²² *p*-Ph-phenoxyacetate, m . 100.5° .³⁵²



$\text{S}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH})_2$, m . 94° ,¹¹⁵² 92° ; ¹¹⁴ diAc., m . 57° .¹¹⁴

$\text{OS}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH})_2$, m . 57° .¹⁴⁶²

$\text{O}_2\text{S}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH})_2$, m . 84° ,¹¹⁵² 83° ,¹³¹¹ 86° .¹⁴⁶²

$\text{S}(\text{CH}_2\text{CH}_2\text{SCHMeCH}_2\text{OH})_2$, m . 59° ; $b_{0.5}$ 210° .¹⁴⁶²

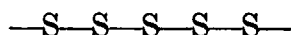
$\text{S}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{OH})_2$, m . 71° .¹¹⁵²



$\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$, m. 111° ,
¹³¹⁹ 107° .¹¹⁵²

$\text{CH}_2(\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH})_2$, m. 75° .¹¹⁵²

$\text{CH}_2(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH})_2$, m. 65° ; ¹¹⁵² diAc., m.
 87° ; diBz., m. 65° .¹³¹⁹



$\text{S}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH})_2$, m. 112.5° .¹¹⁵²

Esters of Thiodiglycol, $\text{S}(\text{CH}_2\text{CH}_2\text{OCOR})_2$

Formate, m. -15.5° ; ²⁹⁵ b_7 $130-7^\circ$, ^{443c}, ^{802a} b_8 134.5° ; d 0/4 1.2525,
d 25/4 1.2275; n 25/D 1.4804.²⁹⁵

Monoacetate, b_{24} $120-1^\circ$, ¹³⁴⁷ b_8 $137-8^\circ$; d 20/4 1.1576, d 25/4
1.1531; n 20/D 1.4879, ¹²¹⁶ n 26/D 1.4728.¹³⁴⁷

Acetate, b_{20} $155-6^\circ$, ⁶²⁸ b_{12} $142-50^\circ$, ^{443c}, ^{802a} b_8 139.5° ; d 0/4
1.1626, d 25/4 1.1382, ²⁹⁵ 1.1366; ³⁰⁷ n 25/D 1.4679, ²⁹⁵ 1.4673.³⁰⁷

Propionate, m. -23° ; ²⁹⁵ b_8 158° ; d 0/4 1.1112, d 25/4 1.0874; n
25/D 1.4648.²⁹⁵

Butyrate, m. -28° ; ²⁹⁵ b_8 172° ; d 0/4 1.0716, d 25/4 1.0491; n
25/D 1.4627.²⁹⁵

i-Valerate, b_8 182° ; d 0/4 1.0384, d 25/4 1.0171; n 25/D 1.4598.²⁹⁵

Caproate, m. 7° , ²⁹⁵ 6.2° ; b_4 187° , ³⁰⁷ b_7 207° ; d 0/4 1.0198, d 25/4
1.0024, ²⁹⁵ 1.0019; n 25/D 1.4620, ³⁰⁷ 1.4627.²⁹⁵

Heptoate, m. 7.5° ; b_2 179° ; d 25/4 0.9857; n 25/D 1.4615.³⁰⁷

Caprylate, m. 27.2° ; b_2 214° .³⁰⁷

Pelargonate, m. 28.6° ; b_2 226° .³⁰⁷

Caprate, m. 46° , ²⁷⁹ 41.5° ; b_2 245° .³⁰⁷

Undecylate, m. 45.3° ; b_2 255° .³⁰⁷

Laurate, m. 57.7° ; b_2 269° .³⁰⁷

Stearate, m. 80° .³⁰⁷

Benzoate, m. 65° .⁵⁰⁸

p-Nitrobenzoate, m. 107.7° .⁹⁰⁵

p-Aminobenzoate, m. 184.5° .⁹⁰⁵

Polysuccinate, m. 44° .³⁰⁷

Polysebacate, m. 76° .³⁰⁷

Polyadipate, m. 60° .³⁰⁷

Polyazellate, m. 56.5° .³⁰⁷

Sulfide-Ethers

- EtSCH₂OEt, b. 134–6°, ^{150a} 135.8°; d 0/4 0.9363, d 25/4 0.9122; n 25/D 1.4432.¹⁴⁴⁶
- PrSCH₂OPr, b. 179.2°; d 0/4 0.9185, d 25/4 0.8960; n 25/D 1.4473.¹⁴⁴⁶
- BuSCH₂OEt, b. 179–81°, ^{1455b} b₃₅ 85°; ^{450c} d 0/4 0.9052, d 25/4 0.8851; n 20/D 1.4502.^{1455b}
- BuSCH₂OBu, b. 220.0°; d 0/4 0.9061, d 25/4 0.8853; n 25/D 1.4500.¹⁴⁴⁶
- PhSCH₂OMe, b₁₈ 113–4°, ^{450a} b₁₂ 108°; d 0/0 1.214, d 16/4 1.046; n 16/D 1.5707.⁸²⁸
- PhCH₂SCH₂OMe, b₁₈ 128°. ^{450a}
- EtSCHMeOMe, b. 130–2°. ³¹⁶
- EtSCHMeOEt, b₅ 33–5°, ^{778.5} b₃₁ 53–4°, b₅₀ 67.5–8.5°; d₂₀ 0.900; n 20/D 1.4428.¹²⁸⁴
- BuSCHMeOEt, b₄ 46–9°. ^{778.5}
- EtSCHMeOBu, b_{2.5} 28–30°, ^{778.5} b₄ 56.1–6.2°; d₂₀ 0.8897; n 20/D 1.4476.¹²⁸⁴
- MeSCH₂CH₂OMe, b. 131°; d 15/4 0.95973, d 20/4 0.95508; n 20/α 1.45178.^{292b}
- MeSCH₂CH₂OPh, b₇ 120°; n 19/D 1.5419.³²⁵
- EtSCH₂CH₂OEt, b₃₅ 78–8.5°; d₂₀ 0.9126; n 20/D 1.4507; sulfoxide, b₈ 112.2°; d₂₀ 1.0354; n 20/D 1.4696.¹²⁸⁴
- EtSCH₂CH₂OBu, b_{3.5} 67.8–8.2°; d₂₀ 0.8897; n 20/D 1.4521; sulfoxide, b₁₅ 112–12.5°; d₂₀ 0.9955; n 20/D 1.4664.¹²⁸⁴
- BuSCH₂CH₂OEt, b₃₀ 105–6°. ^{450a}
- OctSCH₂CH₂OOct, b_{1.7} 170–1°; ^{1175, 1176} n 20/D 1.4616.¹¹⁷⁶
- OctSCH₂CH₂OCH₂CH₂·C₄H₂S·CMe₃-2,5, b_{1.5} 192–8°; n 20/D 1.5008.¹¹⁷⁶
- OctSCHMeCH₂C₆H₄OMe-*p*, b₁ 168–72°. ¹¹²⁷
- CH₂:CHSCH₂CH₂OEt, b₈ 65°; d 20/4 0.9532.³⁴⁰
- CH₂:CHSCH₂CH₂OBu, b_{3.9} 64.5–5°; d 20/4 0.9190; n 20/D 1.4699.¹¹²⁸
- PhSCH₂CH₂OBu, b₁₀ 132°. ^{442.5}
- PhSCH₂CH₂OCH₂CH₂CHMe₂, b₇ 146–50°; ^{1175, 1176} n 20/D 1.5222.¹¹⁷⁶
- PhSCH₂CH₂OPh, b_{0.9} 165–9°; n 20/D 1.5824.¹¹⁷⁶
- PhCH₂SCH₂CH₂OEt, b₁₆ 146°. ¹¹⁹³
- PhCH₂SCH₂CH₂CH₂OEt, b₁₈ 161°. ¹¹⁹³

$\text{PhCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OAm}$, b_3 142–8°.¹¹⁷⁵

$\text{PhCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OPh}$, $b_{0.9}$ 165–9°.¹¹⁷⁵

$\text{MeS}(\text{CH}_2)_{14}\text{OPh}$, m. 46–50°.¹⁰⁷

Multiple Ether-Sulfides

$(\text{MeOCH}_2)_2\text{S}$, b. 152°,¹⁴² b_{17} 53–5°,^{450c} b_{15} 62°; d 0/4 1.0669, d 21.8/4 1.0397; n 21.5/D 1.4575.⁸²⁸

$(\text{BuOCH}_2)_2\text{S}$, b_{37} 98–9°; d 16.5/4 0.8553; n 16.5/D 1.4140.^{1282, 1283}

$(\text{EtOCHMe})_2\text{S}$, $b_{3.2}$ 57.5–8.5°,¹¹²⁶ $b_{4.2}$ 56.5–6.9°,¹²⁸⁴ b_{21} 82–7.5°; ^{911c} d 20/4 0.9425; n 20/D 1.4488,¹²⁸⁴ 1.4474.¹¹²⁶

$(\text{BuOCHMe})_2\text{S}$, $b_{2.2}$ 96.5–6.7°, $b_{3.9}$ 101.5–1.7°; d 20/4 0.9124; n 20/D 1.4520.¹²⁸⁴

$\text{EtOCH}_2\text{CH}_2\text{SCHMeOEt}$, $b_{3.5}$ 65–8°,¹²⁸⁴ $b_{3.3}$ 70.8–1.4°; d 20/4 0.9474; n 20/D 1.4512,¹¹²⁶ 1.4505.¹²⁸⁴

$\text{BuOCH}_2\text{CH}_2\text{SCHMeOBu}$, b_3 107.5–8.5°, $b_{4.2}$ 117.1°; d 20/4 0.9171; n 20/D 1.4537.¹¹²⁶

i- $\text{BuOCH}_2\text{CH}_2\text{SCHMeOBu-}i$, $b_{3.5-4}$ 103–5°; d 20/4 0.9077; n 20/D 1.4486.¹¹²⁶

i- $\text{AmOCH}_2\text{CH}_2\text{SCHMeOAm-}i$, $b_{1.7}$ 102–4°; d 20/4 0.9052; n 20/D 1.4533.¹¹²⁶

$(\text{MeOCH}_2\text{CH}_2)_2\text{S}$, m. –77°; b_{763} 202.2–2.6°,¹¹⁷⁵ b. 201–3.5°; d 4/4 1.0058.^{808a}

$(\text{EtOCH}_2\text{CH}_2)_2\text{S}$, m. –31.5°;¹¹⁷⁵ b. 229°,¹³⁴⁵ 225–6.5°,^{808a} $b_{3.5}$ 83.3–4.4°,¹¹²⁶ b_4 101–2°;³⁴⁰ b_{35} 127–8°;¹¹⁷⁵ d 4/4 0.9672°,^{808a} d 20/4 0.9822,¹³⁴⁵ 0.9550,¹¹²⁶ 0.9658;³⁴⁰ n 20/D 1.4560.¹¹²⁶

$(\text{PrOCH}_2\text{CH}_2)_2\text{S}$, m. –47.5°;¹¹⁷⁵ b. 243–7°,^{808a} $b_{0.4}$ 75–8°,¹¹⁷⁵ b_4 105.8°,¹²⁸⁴ b_{43} 156–7°;^{1175, 1176} d 4/4 0.9439,^{808a} d_{20} 0.9394; n 20/D 1.4533,¹²⁸⁴ 1.4596.^{1175, 1176}

$(i\text{-PrOCH}_2\text{CH}_2)_2\text{S}$, b_6 99–9.5°; d_{20} 0.9258; n 20/D 1.4493.¹⁹⁸⁴

$(\text{BuOCH}_2\text{CH}_2)_2\text{S}$, m. –38°;^{1175, 1176} b. 283–6°,¹¹²⁶ 268–72°,^{808a} b_2 112–3°,¹¹²⁶ $b_{4.5}$ 130–1.0°,¹²⁸⁴ b_{20} 162–4°;¹¹⁷⁵ d 4/4 0.9216,^{808a} d 20/4 0.9223; n 20/D 1.4559,¹¹²⁶ 1.4560.¹²⁸⁴

$(i\text{-BuOCH}_2\text{CH}_2)_2\text{S}$, $b_{3.5}$ 116.2–6.4°;¹¹²⁶ b_5 125–6°; d_{20} 0.9130,¹²⁸⁴ d 20/4 0.9126; n 20/D 1.4510,¹¹²⁶ 1.4506; HgCl_2 , m. 47°.¹²⁸⁴

$(\text{AmOCH}_2\text{CH}_2)_2\text{S}$, m. –33°;^{1175, 1176} $b_{0.3-0.7}$ 120–40°,¹¹⁷⁶ b_{16} 180.5–1°.¹¹⁷⁵

$(i\text{-AmOCH}_2\text{CH}_2)_2\text{S}$, $b_{2.6-2.8}$ 130.2–1°,¹¹²⁶ b_4 144–5°,¹²⁸⁴ b_{20} 210–30°,^{808a} 175–7°;^{1175, 1176} d_{20} 0.9089,¹²⁸⁴ d 20/4 0.961,^{808a} 0.9090; n 20/D 1.4552,¹¹²⁶ 1.4558,^{1175, 1176} 1.4550.¹²⁸⁴

- (HexOCH₂CH₂)₂S, m. -9°; ^{1175, 1176} b_{14.5} 199.5–200.5°.¹¹⁷⁵
 (HepOCH₂CH₂)₂S, m. -5°; ^{1175, 1176} b_{15.5} 217.5–8°.¹¹⁷⁵
 (OctOCH₂CH₂)₂S, m. 15.5°; ^{1175, 1176} b_{0.8} 184–8°,¹¹⁷⁵ b_{2–2.5} 195–8°; d₂₀ 0.8952; n 20/D 1.4610.¹²⁸⁴
 (EtBuCHCH₂OCH₂CH₂)₂S, b_{0.2} 160–5°.¹¹⁷⁵
 (DecOCH₂CH₂)₂S, m. 32.3°; ^{1175, 1176} b_{0.3} 206–10°.¹¹⁷⁵
 (C₆H₁₁OCH₂CH₂)₂S, b₅ 182–4°; d₂₀ 1.0112; n 20/D 1.4982; 0.5 HgCl₂, m. 118°.¹²⁸⁴
 (PhOCH₂CH₂)₂S, m. 54.4°,^{1175, 1176} 54.2°; ⁶²⁶ b_{0.9} 182–5°.^{1175, 1176}
 (*o*-MeC₆H₄OCH₂CH₂)₂S, m. 46.5°.⁶²⁶
 (*p*-MeC₆H₄OCH₂CH₂)₂S, m. 78°.⁶²⁶
 (α-C₁₀H₇OCH₂CH₂)₂S, m. 94.5°.⁶²⁶
 (β-C₁₀H₇OCH₂CH₂)₂S, m. 129°.⁶²⁶
 (4,2-CH₂:CHCH₂(MeO)C₆H₃OCH₂CH₂)₂S, m. 113.5°.⁶²⁶
 [4,2-OCH(MeO)C₆H₃OCH₂CH₂]₂S, m. 131.5°.⁶²⁶
 (2,4,6-Br₃C₆H₂OCH₂CH₂)₂S, m. 118.5°.⁶²⁶
 (C₄H₃O·CH₂OCH₂CH₂)₂S, b_{0.8} 162–3°; n 20/D 1.4935.¹¹⁷⁶
 AmOCH₂CH₂SCH₂CH₂OPh, b₃ 142–8°; n 20/D 1.5148.¹¹⁷⁶
 β-C₁₀H₇OCH₂CH₂SCH:CHOC₁₀H₇-β, m. 129.5°.⁸³⁰
 2,4,6-Br₃C₆H₂OCH₂CH₂SCH₂CH₂CH₂OC₆H₂Br₃-2,4,6, m. 80°.³⁵²
 2,4,6-Br₃C₆H₂OCH₂CH₂SCH₂CHMeOC₆H₂Br₃-2,4,6, m. 126°.³⁵²
 2,4,6-Br₃C₆H₂OCH₂CH₂SCHMeCH₂OC₆H₂Br₃-2,4,6, m. 127°.³⁵²
 (MeOCH₂CH₂CH₂)₂S, m. 52°.^{111a}
 (PhOCH₂CH₂CH₂)₂S, m. 45°.^{111a}
 (*p*-MeC₆H₄OCH₂CH₂CH₂)₂S, m. 52°.^{111a}
 (MeOCH₂CH₂CH:CHCH₂)₂S, b₁₃ 163°,¹¹³³ b₁₀ 161°; ¹¹³⁴ d 20/4 0.9763; n 20/D 1.4920,¹¹³³ 1.4940.¹¹³⁴
 (EtOCH₂CH₂CH:CHCH₂)₂S, b₁₂ 172–5°; d 20/4 0.9460; n 20/D 1.4830.¹¹³⁴
 (BuOCH₂CH₂CH:CHCH₂)₂S, b₆ 185–7°; d 20/4 0.9204; n 20/D 1.4770.¹¹³⁴
 MeS(CH₂)₁₄OCH₂CH₂OBu, m. 60–8°.¹⁰⁷
 MeS(CH₂)₁₄OCH₂CH₂OPh, m. 46–50°.¹⁰⁷
 (*p*-MeOC₆H₄CH₂CH₂)₂S, b_{0.3} 160–2°.^{694b, 763}
 (2-EtOC₆H₁₀)₂S, b₁₅ 165°; d₂₅ 1.033; n 25/D 1.5057.^{1005b}

TABLE 1.7
Melting Points ($^{\circ}\text{C}$) of $S(\text{CH}_2\text{CH}_2\text{OR})_2$ ¹¹⁷⁵

Me	-77 $^{\circ}$	C_7H_{15}	-5 $^{\circ}$	$\text{C}_{13}\text{H}_{27}$	44 $^{\circ}$
Et	-31.5 $^{\circ}$	C_8H_{17}	15.5 $^{\circ}$	$\text{C}_{14}\text{H}_{29}$	54 $^{\circ}$
Pr	-47.5 $^{\circ}$	C_9H_{19}	17 $^{\circ}$	$\text{C}_{15}\text{H}_{31}$	53 $^{\circ}$
Bu	-38 $^{\circ}$	$\text{C}_{10}\text{H}_{21}$	32.3 $^{\circ}$	$\text{C}_{16}\text{H}_{33}$	60.3 $^{\circ}$
Am	-33 $^{\circ}$	$\text{C}_{11}\text{H}_{23}$	32.5 $^{\circ}$	$\text{C}_{17}\text{H}_{35}$	60 $^{\circ}$
Hex	-9 $^{\circ}$	$\text{C}_{12}\text{H}_{25}$	44.5 $^{\circ}$	$\text{C}_{18}\text{H}_{37}$	66.5 $^{\circ}$

Sulfide-Acetals and Sulfide-Ortho-Esters

ACETALS

$\text{MeSCH}_2\text{CH}(\text{OEt})_2$, b. 168–70 $^{\circ}$, b_{25} 91 $^{\circ}$.¹⁰²¹
 $\text{PhSCH}_2\text{CH}(\text{OEt})_2$, b. 273 $^{\circ}$.⁴⁸
 $\text{PhCH}_2\text{SCH}_2\text{CH}(\text{OMe})_2$, b_{3-4} 90–5 $^{\circ}$,^{1075.5} b_6 140–1 $^{\circ}$; d 22/4 1.0757; n 22/D 1.5303,⁵³⁶ n 30/D 1.5299.^{1075.5}
 $\text{PhCH}_2\text{SCH}_2\text{CH}(\text{OEt})_2$, b_{25} 178 $^{\circ}$,¹⁰²¹ b_{30} 192–5 $^{\circ}$,⁶⁹⁰ $b_{0.5}$ 114–20 $^{\circ}$.⁶⁴⁵
 $\text{PhCH}_2\text{SCMe}_2\text{CH}(\text{OEt})_2$, b_{13} 167–8 $^{\circ}$; n 18/D 1.5155.¹³⁷⁴
 $\text{MeSCH}_2\text{CH}_2\text{CH}(\text{OMe})_2$, $b_{0.9}$ 73 $^{\circ}$.²⁹⁴
 $\text{MeSCH}_2\text{CH}_2\text{CH}(\text{OEt})_2$, $b_{0.7}$ 68–74 $^{\circ}$.²⁹⁴
 $\text{EtSCH}_2\text{CH}_2\text{CH}(\text{OEt})_2$, b_9 94–7 $^{\circ}$.^{1211d}
 $\text{S}[\text{CH}_2\text{CH}(\text{OMe})_2]_2$, b_1 85 $^{\circ}$; n 25/D 1.4569.^{1075.5}
 $\text{S}[\text{CH}_2\text{CH}(\text{OEt})_2]_2$, b_{750} 280 $^{\circ}$, b_{11} 143–7 $^{\circ}$.⁴⁶⁹
 $\text{S}[\text{CH}_2\text{CH}_2\text{CH}(\text{OEt})_2]_2$, $b_{0.27}$ 130–2 $^{\circ}$.⁸⁹⁰

ORTHO-ESTERS

$\text{MeSCH}_2\text{C}(\text{OEt})_3$, b_{30} 78–80 $^{\circ}$.⁷⁷⁹
 $\text{MeSCH}_2\text{CH}_2\text{C}(\text{OMe})_3$, b_1 51–2 $^{\circ}$.²⁹⁴
 $\text{MeSCH}_2\text{CH}_2\text{C}(\text{OEt})_3$, $b_{0.8}$ 71–2 $^{\circ}$.²⁹⁴

Aromatic Sulfide-Ethers

$o\text{-MeSC}_6\text{H}_4\text{OMe}$, m. 19 $^{\circ}$; ¹¹⁰⁹ b . 238 $^{\circ}$,⁶⁶⁷ 237 $^{\circ}$.^{532a}
 $p\text{-MeSC}_6\text{H}_4\text{OMe}$, m. 26 $^{\circ}$; ^{1506b} b . 239–40 $^{\circ}$,^{532a} b_4 99 $^{\circ}$; d 25/4 1.1069; n 25/D 1.5764.¹³⁴⁰
 $p\text{-MeSC}_6\text{H}_4\text{OEt}$, m. 21.5 $^{\circ}$; b . 250–1 $^{\circ}$,⁶⁷⁶ b_5 98–100 $^{\circ}$; d 25/4 1.0693; n 25/D 1.5618.¹³⁴⁰

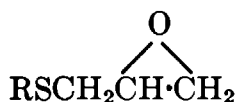
- p -EtSC₆H₄OMe, b_5 103°; d 25/4 1.0674; n 25/D 1.5600.¹³⁴⁰
 EtSC₆H₄OEt, *ortho*, b . 248–50°; ^{532a} *para*, b 259–60°, ^{532a} b_6 110–2°.¹³⁴⁰
 p -PrSC₆H₄OMe, b_5 110°; d 25/4 1.0424; n 25/D 1.5545.¹³⁴⁰
 p -BuSC₆H₄OMe, b_5 120°; d 25/4 1.0303; n 25/D 1.5445.¹³⁴⁰
 p -AmSC₆H₄OMe, b_5 127°; d 25/4 1.0149; n 25/D 1.5380.¹³⁴⁰
 p -HexSC₆H₄OMe, b_5 142°; d 25/4 0.9975; n 25/D 1.5315.¹³⁴⁰
 o -PhSC₆H₄OMe, b_{11} 196°, ^{932b} b_3 150–2°, ⁶⁴⁸ b_{4-5} 164–6°.¹¹²⁹
 m -PhSC₆H₄OMe, b_4 156°.⁶⁴⁸
 p -PhSC₆H₄OMe, b_{13} 194–5°, ⁶⁴⁸ b_{12} 188°, ^{932b} 180–5°, ^{443a}, ^{694a} b_{11} 178–80°, ⁸³⁴ b_6 161–3°.⁶⁴⁸
 o -PhSC₆H₄OPh, b_3 150–2°.⁶⁴⁸
 p -MeC₆H₄SC₆H₄OMe- p , m . 46°; b_4 181–2°.⁶⁴⁸
 p -PhCH₂SC₆H₄OMe, m . 46°.^{1351a}, ^{1351b}
 p -PhCH₂SC₆H₄OEt, m . 44°.^{1351a}, ^{1351b}
 MeSC₆H₃(Me)OMe-2,4, d 25/4 1.0867; n 25/D 1.5712.¹³⁴¹
 MeSC₆H₃(OMe)Me-2,5, b_{39} 145–50°.¹⁵⁰¹
 EtSC₆H₃(Me)OMe-2,4, d 25/4 1.0554; n 25/D 1.5576.¹³⁴¹
 PrSC₆H₃(OMe)Me-4,2, d 25/4 1.0362; n 25/D 1.5496.¹³⁴¹
 BuSC₆H₃(OMe)Me-4,2, d 25/4 1.0182; n 25/D 1.5436.¹³⁴¹
 AmSC₆H₃(OMe)Me-4,2, d 25/4 1.0086; n 25/D 1.5399.¹³⁴¹
 PhCH₂SC₆H₃(OCH₂Ph)Me-2,5, m . 85°.¹⁵⁰¹
 MeSC₆H₃(OMe)₂-2,4, m . 33–7°.¹¹⁰⁸
 i -AmSC₆H₃(OMe)₂-2,5, m . 33°; $b_{0.5}$ 153–5°, ^{228a}
 p -MeSC₆H₄CHPhOEt, b_{15} 206–8°; d 22/4 1.0891; n 20/D 1.5910.⁷⁴
 p -MeC₆H₄SCH₂C₆H₄OMe- p , m . 67°.⁷⁵
 p -MeC₆H₄SCHMeC₆H₄OMe- p , m . 58°.⁷⁵
 p -MeC₆H₄SCHPhC₆H₄OPh- p , m . 56°.⁷⁵
 p -MeC₆H₄SCHPhC₆H₃(OMe)₂-3,4, m . 75°.⁷⁵
 p -MeOC₆H₄CH₂CH(SMe)C₆H₄OMe- p , m . 154.5°.¹⁰⁵⁷
 p -MeOC₆H₄CH(OMe)CH(SMe)C₆H₄OMe- p , m . 191°.¹⁰⁵⁷
 p -MeC₆H₄CH(SMe)CH(SMe)C₆H₄OMe- p , m . 190°.¹⁰⁵⁷
 p -MeOC₆H₄C(SMe):C(SMe)C₆H₄OMe- p , m . 196°.¹⁰⁵⁷
 $(o$ -MeOC₆H₄)₂S, m . 73°; b_{10} 252–3°.^{932a}, ^{932b}
 $(m$ -MeOC₆H₄)₂S, b_{10} 214–5°.^{932b}
 $(p$ -MeOC₆H₄)₂S, m . 46°, ⁸⁷¹, ^{932b} 45°; b_2 166–70°, ⁶¹⁵ b_{12} 215°.^{932b}

- $(p\text{-EtOC}_6\text{H}_4)_2\text{S}$, m. 55° .⁸⁷¹
 $1\text{-MeSC}_{10}\text{H}_6\text{OMe-4}$, m. 63° .¹⁵¹⁷
 $1\text{-MeSC}_{10}\text{H}_6\text{OMe-5}$, m. 134° .¹¹⁶⁴
 $2\text{-MeSC}_{10}\text{H}_6\text{OMe-6}$, m. 106° .¹⁵⁰⁵
 $\alpha\text{-(}\beta\text{-MeOC}_{10}\text{H}_6)_2\text{S}$, m. 184° ,^{1042b} 195° .⁸⁷¹
 $\beta\text{-(}\alpha\text{-MeOC}_{10}\text{H}_6)_2\text{S}$, m. 135° .⁸⁷¹
 $\alpha\text{-(}\beta\text{-EtOC}_{10}\text{H}_6)_2\text{S}$, m. 189° ,¹⁰⁶⁰ 195° .⁸⁷¹
 $\beta\text{-(}\alpha\text{-EtOC}_{10}\text{H}_6)_2\text{S}$, m. 153° .⁸⁷¹
 $9,10\text{-Dimethoxy-9,10-diphenyl-9,10-dihydroanthracenyl-1-methyl sulfide}$, m. 220° .¹⁰⁷⁰

Multiple Sulfide-Ethers

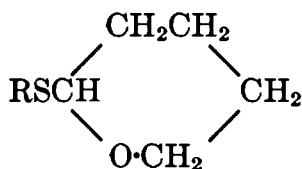
- $(t\text{-BuSCH}_2\text{CH}_2)_2\text{O}$, b_{11} $143\text{--}7^\circ$.¹³⁰⁴
 $(\text{AmSCH}_2\text{CH}_2)_2\text{O}$, b_1 $155\text{--}65^\circ$.⁸²
 $(\text{C}_{10}\text{H}_{21}\text{SCH}_2\text{CH}_2)_2\text{O}$, m. 44° .³⁸³
 $(\text{C}_{12}\text{H}_{25}\text{SCH}_2\text{CH}_2)_2\text{O}$, m. 32° ,¹³²⁵ 56° .³⁸³
 $(\text{PhSCH}_2\text{CH}_2)_2\text{O}$, b_{16} $257\text{--}8^\circ$; d $15/4$ 1.1561 .³²⁶
 $(\text{EtOCH}_2\text{CH}_2\text{SCH}_2)_2$, b_{20} $175\text{--}90^\circ$; d $4/4$ 1.0450 .^{808a}
 $(\text{EtOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2)_2\text{O}$, b_{13} $202\text{--}6^\circ$; d_{20} 1.0504 .^{808a}
 $(\text{PrOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2)_2\text{O}$, b_{20} $228\text{--}30^\circ$; 1175 , 1176 d $20/4$ 1.0226 ; 1175 n $20/D$ 1.4898 .^{1175, 1176}
 $(\text{PhOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2)_2\text{O}$, m. 60° .^{1482a}
 $(\text{PhOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2)_2\text{S}$, m. 59° .¹⁴⁶²
 $(\alpha\text{-C}_{10}\text{H}_7\text{OCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2)_2\text{S}$, m. 56° .¹⁴⁶²
 $(\beta\text{-C}_{10}\text{H}_7\text{OCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2)_2\text{S}$, m. 122° .¹⁴⁶²
 $(\text{MeSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2)_2\text{O}$, m. 35.5° .²¹¹
 $(\text{MeSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2)_2\text{S}$, m. 42° .²¹²
 $(\text{PhSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2)_2\text{S}$, m. 44° .²¹²
 $(p\text{-MeSC}_6\text{H}_4)_2\text{O}$, m. 52° .¹³⁹³
 $(p\text{-EtSC}_6\text{H}_4)_2\text{O}$, m. 57° .¹³⁹³
 $o\text{-MeSC}_6\text{H}_4\text{SO}_2\text{C}_6\text{H}_4\text{OMe-}o$, m. 197° .^{500a}
 $1,3\text{-(MeS)}_2\text{C}_6\text{H}_2(\text{OMe})_2\text{-4,6}$, m. 86° .¹¹⁰⁸
 $(\text{MeSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2)_2\text{O}$, m. 45° .²¹²
 $(\text{PhSCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2)_2\text{O}$, m. 50° .²¹²
 $[\text{PhS}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{O})_2\text{CH}_2\text{CH}_2]_2\text{S}$, m. 55° .²¹²
 $[\text{PhS}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{O})_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2]_2\text{O}$, m. 57° .²¹²

TABLE 2.7

Alkylmercaptopropylene Oxide

R	B.p.(°C)	mm. pressure	d 25/4	n 25/D	Ref.
Methyl	85°	7.0	1.0583	1.4817	1391
Ethyl	42°	3.5	1.0170	1.4757	1391
Propyl	45°	2	0.9905	1.4730	1391
Butyl	132°	70	0.9716	1.4723	1391
Amyl	68°	1.6	0.9544	1.4707	1391
Hexyl	70°	0.7	0.9471	1.4702	1391
Dodecyl	210–20°	2	—	—	783
<i>p</i> -MeC ₆ H ₄	107.5°	0.2			331a

TABLE 3.7

Alkylmercaptopentamethylene Oxide ^{778d}

	B.p.(°C)		B.p.(°C)
Methyl	b ₅ 47–8°	<i>i</i> -Propyl	b _{2.5} 48°
Ethyl	b ₂ 42°	Hexyl	b _{2.5} 84–6°
Propyl	b ₃ 55–8°	Benzyl	b ₂ 86–7°

Hydroxy-Ether-Sulfides

EtSCH₂CH(OH)CH₂OMe, b₉ 92.5–2.8°; d 25/4 1.0322; n 25/D 1.4734.¹¹⁸¹

EtSCH₂CH(OH)CH₂OEt, b₄₀ 136–7°; d 25/4 1.0037; n 25/D 1.4679.¹¹⁸¹

EtSCH₂CH(OH)CH₂OPr, b₂ 78–8.5°; d 25/4 0.9837; n 25/D 1.4661.¹¹⁸¹

- EtSCH₂CH(OH)CH₂OBu, *b*₂ 85.5–6.5°; *d* 25/4 0.9695; *n* 25/D 1.4652.¹¹⁸¹
- PrSCH₂CH(OH)CH₂OMe, *b*₂ 65–6°; *d* 25/4 1.0095; *n* 25/D 1.4719.¹¹⁸¹
- PrSCH₂CH(OH)CH₂OEt, *b*_{1.5} 69.5–70°; *d* 25/4 0.9850; *n* 25/D 1.4670.¹¹⁸¹
- PrSCH₂CH(OH)CH₂OPr, *b*_{1.5} 81–1.5°; *d* 25/4 0.9676; *n* 25/D 1.4641.¹¹⁸¹
- PrSCH₂CH(OH)CH₂OBu, *b*_{1.5} 90.5–1.5°; *d* 25/4 0.9578; *n* 25/D 1.4639.¹¹⁸¹
- MeSCH₂C₆H₃(OMe)OH-3,4, *b*_{7.5} 144°; *d*₁₈ 1.1803.¹²⁸
- EtSCH₂CH(OH)C₆H₄OMe-*p*, *b*_{0.5} 125–6°.¹¹¹⁷
- MeSCH₂CH(OH)C₆H₃(OMe)₂-3,4, *m*. 71°.¹¹¹⁷
- p*-MeOC₆H₄SCH₂CH₂OH, *m*. 41°.^{68a}
- MeOCH₂CH₂SCH₂CH₂OH, *b*₁₅ 112°.⁵⁷⁵
- EtOCH₂CH₂SCH₂CH₂OH, *b*. 238–46°,^{808a} *b*₄ 117.5°,³⁴⁰ *b*₁₃ 115–7°,⁵⁷⁵ *b*₂ 93–4°;³⁵² *d*₂₀ 1.0474,^{808a} *d* 25/4 1.0428; *n* 25/D 1.4795;³⁵² viscosity at 25° 5.44.³⁵²
- PrOCH₂CH₂SCH₂CH₂OH, *b*₁₂ 128–9°.⁵⁷⁵
- i*-PrOCH₂CH₂SCH₂CH₂OH, *b*₁ 94°; *d* 25/4 1.0125; *n* 25/D 1.4735;³⁵² viscosity at 25° 6.97.³⁵²
- BuOCH₂CH₂SCH₂CH₂OH, *b*_{1.5} 96–8°,³⁵² *b*₂₀ 152–4°,¹¹⁷⁵ *b*₁₀ 136°;¹¹⁵⁸ *d* 25/4 0.997,³⁵² 1.0160; *n* 25/D 1.5033,¹¹⁵⁸ 1.4746; viscosity at 25° 8.10.³⁵²
- CH₂:CHCH₂OCH₂CH₂SCH₂CH₂OH, *b*_{0.006} 96°.³⁵²
- (MeO)₂CHCH₂SCH₂CH₂OH, *b*_{0.63} 90°; *n* 20/D 1.4810.^{1074.5}
- (EtO)₂CHCH₂SCH₂CH₂OH, *b*_{1.6} 112°; *n* 20/D 1.4710.^{1074.5}
- (EtO)₂CHCH₂SCH₂CHMeOH, *b*_{0.55} 92–3°; *n* 20/D 1.4654.^{1074.5}
- (EtO)₂CHCH₂SCH₂CMe₂OH, *b*_{0.3} 95°; *n* 20/D 1.4650.^{1074.5}
- (EtO)₂CHCHMeSCH₂CH₂OH, *b*_{0.5} 87°.¹⁴⁶²
- S(CH₂CH₂OCH₂CH₂OH)₂, *b*_{1.5} 180°.²¹²
- HSCH₂CH₂OCH₂CH₂SCH₂CH₂OH, *b*_{1.5} 60°.²¹²
- O(CH₂CH₂SCH₂CH₂OH)₂, *m*. 31.2°,³⁵² 32°,^{1482a} 30°;²¹² *b*_{2.5} 215°;^{1482a} *d* 35/4 1.1779; viscosity at 35° 54.8.³⁵²
- O(CH₂CH₂SCHMeCH₂OH)₂, *b*₅ 217–8°.¹⁴⁶²
- O(CH₂CH₂CH₂SCH₂CH₂OH)₂, *d* 25/4 1.1400; *n* 25/D 1.5275; viscosity at 25° 142; *p*-Ph-phenoxyacetate, *m*. 60°.³⁵²
- O(CH₂CH₂SCH₂CH₂OCH₂CH₂OH)₂, *b*₁ 230°.²¹²
- S(CH₂CH₂OCH₂CH₂SCH₂CH₂OH)₂, *m*. 49°.²¹²
- O(CH₂CH₂SCH₂CH₂OCH₂CH₂SCH₂CH₂OH)₂, *m*. 58.5°.²¹²

$S(CH_2CH_2OCH_2CH_2SCH_2CH_2OCH_2CH_2SCH_2CH_2OH)_2$, m.
61°. ²¹²

$O(CH_2CH_2SCH_2CH_2OCH_2CH_2SCH_2CH_2OCH_2CH_2SCH_2CH_2OH)_2$, m. 62.5°. ²¹²

α -Chlorosulfides

$MeSCH_2Cl$, b. 110°, ¹⁵³ 105–7°; ^{1430a} b_{750} 107.1°, b_{100} 50–2°; n
20/D 1.4967. ¹³⁹⁷

$EtSCH_2Cl$, b. 128–31°, ^{150a} 128–30°, ^{732a} 127–9°. ^{1430a}

$PrSCH_2Cl$, b. 149–50°, ^{1430a} b_{80} 58°. ¹⁵²

i- $PrSCH_2Cl$, b. 138–9°, ^{1430a} b_{42} 56–8°. ¹⁵²

$BuSCH_2Cl$, b_{18} 64–6°. ¹⁴²⁸, ^{1430a}

i- $BuSCH_2Cl$, b. 160–1°. ¹⁴²⁸, ^{1430a}

s- $BuSCH_2Cl$, b_{11} 58–9°. ¹⁴²⁸, ^{1430a}

t- $BuSCH_2Cl$, b_{12} 57–8°, ¹⁴²⁸, ^{1430a} b_{18} 48–9°; n 20/D 1.4816. ¹³⁹⁷

$AmSCH_2Cl$, b. 172–6°. ¹⁴²⁸, ^{1430a}

s- $AmSCH_2Cl$, b_{13} 73–6°. ¹⁴²⁸, ^{1430a}

i- $AmSCH_2Cl$, b_{22} 105–6°, ¹⁴²⁸ b_{30} 91–3°. ^{1430a}

$HexSCH_2Cl$, b_{22} 105–6°. ^{1430a}

$Et_2CHCH_2SCH_2Cl$, b_9 87–8°. ^{1430a}

$HexCHMeSCH_2Cl$, $b_{1.5}$ 50–3°, ¹⁴²⁸ b_2 78–80°. ^{1430a}

$C_6H_{11}SCH_2Cl$, $b_{13.5}$ 101–3°. ¹⁴²⁸, ^{1430a}

$CH_2:CHCH_2SCH_2Cl$, b_{15} 52–5°. ¹⁴²⁸, ^{1430a}

$PhSCH_2Cl$, b_9 99–102°, ⁴⁴² b_{12} 98°. ¹⁵²

$PhCH_2SCH_2Cl$, b_{15} 120–5°, ⁷⁹ b_{25} 136–9°, ¹⁵² b_{13} 122°. ^{150.5}

p- $MeC_6H_4SCH_2Cl$, b_{15} 126–9°. ⁷⁹

p- $ClC_6H_4SCH_2Cl$, m. 21.5°; ⁷⁹ b_{17} 128–30°, ¹⁵² b_{22} 147.5–8.5°, ⁷⁹
 b_{12} 132°. ⁴⁴²

4,6- $Cl_2C_6H_2(SCH_2Cl)_2$ -1,3, m. 88.5°. ⁷⁹

$MeSCHMeCl$, b_{100} 51–5°, ¹⁵² b_{110} 59°. ¹⁵³

$EtSCHMeCl$, b_{20} 50–4°, ^{732a} b_{24} 39°, ¹⁵³ b_{40} 56°. ¹⁵³

i- $PrSCHMeCl$, b_{12} 43–7°, $b_{12.5}$ 43°. ¹⁴²⁹, ^{1430b}

$BuSCHMeCl$, b_3 43–5°, ^{1430b} b_{13} 65–7°. ¹⁴²⁹

$AmSCHMeCl$, $b_{10.5}$ 50–3°. ¹⁴²⁹, ^{1430b}

$CH_2:CHSCHMeCl$, b_{15} 36°; d 15/4 1.078. ⁷³

$EtSCHEtCl$, b_{18} 45–55°. ^{732a}

$EtSCHMe_2Cl$, b_{20} 45–60°. ^{732a}

$EtSCHPrCl$, b_{10} 55–62°. ^{732a}

$EtSCH(CHMe_2)Cl$, b_{10} 48–52°, b_{19} 60–5°. ^{732a}

$MeSCHPhCl$, b_{15} 121–2°, ¹⁵³ b_{15} 116–7°. ¹⁵²

EtSCHPhCl, b_{13} 137–9°. ^{732a}
 PhCH₂SCHPhCl, $b_{0.5}$ 160°. ¹⁵²
 MeSCHCl₂, m. –36°; b_{742} 137.0°, ¹³⁹⁷ b_{15} 48–9°, ¹⁵³ b_{50} 60.1°; d 20/4 1.3702; n 20/D 1.5159. ¹³⁹⁷
t-BuSCHCl₂, b_2 46–7°; d 20/4 1.2132; n 20/D 1.5056. ¹³⁹⁷
 PhSCHCl₂, b_{15} 117–8°. ¹⁵³
 MeSCCl₃, m. –5°; b_{750} 146.0°, ¹³⁹⁷ b_{13} 59–60°, ¹⁵³ b_{50} 67.7°; d 20/4 1.4907; n 20/D 1.5219. ¹³⁹⁷
 EtSCCl₃, b_{10} 85°. ¹²²⁵
t-BuSCCl₃, m. 54.5°; b_4 72.5–5°, b_{11} 90.2°. ¹³⁹⁷
 PhSCCl₃, m. 36°, ¹⁵³ 25.5°; ^{1093.5} b_{14} 123–5°, ¹⁵³ b_{10} 135°. ¹²²⁵
p-MeC₆H₄SCCl₃, m. 23°; b_{15} 150°. ¹⁵⁰⁸
p-ClC₆H₄SCCl₃, m. 61°. ^{1093.5}
 2,4-Cl₂C₆H₃SCCl₃, m. 45.5°. ^{1093.5}
m-C₆H₄(SCCl₃)₂, m. 106°. ^{467b}, ¹⁵¹²
p-C₆H₄(SCCl₃)₂, m. 149°. ^{467b}
p,p'-(·C₆H₄SCCl₃)₂, m. 195°. ¹⁵⁰⁴

β-Chlorosulfides

MeSCH₂CH₂Cl, b . 140°, ⁷⁸² b_{744} 138.0–8.7°, ¹¹⁴⁸ b_{20} 44°, ^{780a} 55–7°, ⁴⁶² b_{30} 55–6°, ^{780a}, ⁷⁸² 51–5°; ¹³³⁸ d 20/4 1.1155, ¹¹⁴⁸ 1.1226; ^{780a} n 20/D 1.4908, ⁴⁶² n 30/D 1.4902. ^{780a}
 EtSCH₂CH₂Cl, b . 157°, ³⁶⁷, ^{956c}, ^{989d} 156.5°, ^{989c} 154–5°, ⁵⁷⁵ b_{20} 59°, ^{1011b} b_{37} 69–73°, ⁶⁸⁹ b_{40} 71–2°, ¹¹⁴⁸ b_{47} 63–5°, ^{350a} b_{51} 70°; ^{989d} d 20/4 1.0664, ¹¹⁴⁸ d 22/4 1.0701, ^{989c} d 25/4 1.0758, ^{1011b} 1.0663; ^{350a}, ^{989c}, ¹¹⁴⁸ n 20/D 1.4878, ¹¹⁴⁸ 1.4898; ^{1011b} parachor 277.5 at 22.5°. ^{989c}, ^{1011b}
 PrSCH₂CH₂Cl, b_{22} 74–5°, ⁵⁷⁵ b_2 43–5°; d 25/4 1.0349. ^{350a}
 BuSCH₂CH₂Cl, b_1 58–9°, ^{350a} b_3 73–5°, ⁸⁸⁶ b_6 68°, ^{1455a} b_{20} 89°, ⁵⁷⁵ b_{26} 96°, ¹²⁷ 98–100°; ⁶⁸⁹ d 0/4 1.0332, ^{1455a} d 25/4 1.0122, ^{350a} 1.0072; n 20/D 1.4825. ^{1455a}
i-BuSCH₂CH₂Cl, b_{25} 84–5°. ²²⁰
s-BuSCH₂CH₂Cl, b_{40} 98–100°. ⁶⁸⁹
t-BuSCH₂CH₂Cl, m. –49°; b_{30} 81–2°, ^{350d} b_{32} 85–8°, ⁶⁸⁹ b_{10} 53–6°; ¹³⁰⁴ d_{25} 1.0001. ^{350d}
i-AmSCH₂CH₂Cl, b_1 68°; d 25/4 0.9899. ^{350a}
 HepSCH₂CH₂Cl, b_{25} 139–40°. ⁵⁷⁵
 OctSCH₂CH₂Cl, b_4 117–9.5°; n 25/D 1.4741. ^{466c}
t-OctSCH₂CH₂Cl, b_1 73–8°; n 25/D 1.4869. ^{466c}
 NonSCH₂CH₂Cl, b_{20} 164–6°. ⁵⁷⁵

- $\text{DecSCH}_2\text{CH}_2\text{Cl}$, b_1 118–21°; n 25/D 1.4750.^{466c}
 $\text{C}_{11}\text{H}_{23}\text{SCH}_2\text{CH}_2\text{Cl}$, b_{14} 175–9°.⁵⁷⁵
 $\text{C}_{12}\text{H}_{25}\text{SCH}_2\text{CH}_2\text{Cl}$, $b_{1-1.5}$ 152–3°; n 25/D 1.4740.^{466c}
 $t\text{-C}_{14}\text{H}_{29}\text{SCH}_2\text{CH}_2\text{Cl}$, $b_{1.4}$ 140–2°; b_1 121–4°.^{466c}
 $\text{C}_{18}\text{H}_{37}\text{SCH}_2\text{CH}_2\text{Cl}$, m . 35°.^{466c}
 $\text{H}_2\text{C:CHSCH}_2\text{CH}_2\text{Cl}$, b_{764} 152.5°, b_{50} 71–2°.³⁴⁰
 $\text{H}_2\text{C:CHCH}_2\text{SCH}_2\text{CH}_2\text{Cl}$, b_{12} 67.5–9°, 68°, ¹²⁷⁶ b_{14} 66–7°; ¹¹⁴⁸ d 20/4 1.0750, ¹²⁷⁶ 1.0761; n 20/D 1.5071.¹¹⁴⁸
 $\text{C}_6\text{H}_{11}\text{SCH}_2\text{CH}_2\text{Cl}$, b . 133°.⁶⁵⁹
 $\text{PhSCH}_2\text{CH}_2\text{Cl}$, b . 245°, ¹³²³ $b_{0.64}$ 88–9°, ^{350a} b_4 100–1°, ^{781b} b_6 127–9°, ⁶⁸⁹ b_7 107°, ⁴⁸⁰ b_9 115–7°, ⁷⁷² 114–5°, ¹¹²¹ b_{13} 122°, ⁸³⁴ b_{15} 121–2°, ^{21a} b_{26} 144.5°; ¹³²³ d 20/4 1.1799, ¹¹⁰ 1.1808, ^{781b} d 23.5/4 1.1820, ⁴⁸⁰ d 25/4 1.1769; ^{350a} n 20/D 1.5838, ^{781b} 1.5837, ¹¹⁰ n 25/D 1.5828.^{21a}
 $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{Cl}$, b_1 114–6°, ¹⁰⁸³ 113–5°, ^{350a} b_{12} 137–8°, ⁵⁷⁵ b_{18} 145°; ¹¹⁴⁸ d 25/4 1.1479, ^{350a} 1.1477; n 20/D 1.571.¹¹⁴⁸
 $p\text{-MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{Cl}$, b . 255–7°, ⁵⁰⁶ b_1 100–1°, ⁸⁸⁶ b_{17} 139.5–40.5°, ⁸³⁴ b_{20} 150–2°, ⁵⁵⁴ b_{22} 150°, ⁵⁰⁶ 147–51°.¹²⁹⁸
 $2,4,6\text{-Me}_3\text{C}_6\text{H}_2\text{SCH}_2\text{CH}_2\text{Cl}$, m . 44°.^{66a}
 $\beta\text{-C}_{10}\text{H}_7\text{SCH}_2\text{CH}_2\text{Cl}$, m . 53°.^{66a}
 $2\text{-C}_4\text{H}_9\text{OCH}_2\text{SCH}_2\text{CH}_2\text{Cl}$, b_5 128°; d 30/4 1.1863; n 30/D 1.5200.⁵⁵⁹
 $3\text{-C}_4\text{H}_9\text{SCH}_2\text{CH}_2\text{Cl}$, b_8 123°.⁶⁷⁵
 $\text{MeSCH}_2\text{CHMeCl}$, b_{17} 47–8°, ^{924.5} b_{37} 67°; d 20/4 1.076; n 20/D 1.4905, ⁶⁸⁴ 1.4851.^{924.5}
 $\text{MeSCH}_2\text{CHPrCl}$, b_{20} 84–6°; d 20/4 1.0090; n 20/D 1.4860.⁵⁶⁹
 $\text{MeSCH}_2\text{CHPhCl}$, b_{13} 137°.^{202.5}
 $\text{EtSCH}_2\text{CHMeCl}$, b_{12} 52–5°, ^{1430c} b_{17} 60°; d 25/4 1.0265; ^{850a} n 20/D 1.4780.⁵²³
 $\text{EtSCHMeCH}_2\text{Cl}$, n 20/D 1.4782.⁵²³
 $\text{EtSCH}_2\text{CHPhCl}$, b_{14} 146°.^{202.5}
 $\text{PrSCH}_2\text{CHPhCl}$, b_{11} 148°.^{202.5}
 $i\text{-PrSCH}_2\text{CHPhCl}$, b_{12} 147°.^{202.5}
 $\text{BuSCH}_2\text{CHPhCl}$, b_{13} 158°.^{202.5}
 $\text{CH}_2\text{:CHSCH}_2\text{CHPhCl}$, b_8 135°.^{202.3}
 $p\text{-MeC}_6\text{H}_4\text{SCHMeCHMeCl}$, *cis*, b_3 122–3°; n 26/D 1.5486; *trans*, b_4 131–2°; n 26/D 1.5530; sulfone, m . 56°.^{768.5}
 $\text{H}_2\text{C:CHSCHClCH}_2\text{Cl}$, See Chlorinated Ethyl-Vinyl and Divinyl Sulfides
 $\text{PhSCCl}_2\text{CHCl}_2$, b_{20} 175–82°.³³⁴
 EtSCHClCCl_3 , b_{13} 101–2°.^{150.5}

Other Chlorosulfides

MeSCH₂CH₂CH₂Cl, *b*₂₀ 71.2°; *d* 20/4 1.0845; *n* 30/D 1.4833.^{780a}
 EtSCH₂CH₂CH₂Cl, *b*₁₇ 73°, ^{1211a} 72°, ^{350a} *b*₁₈ 58–72°; ⁸¹⁰ *d* 25/4
 1.0427.^{350a}

PrSCH₂CH₂CH₂Cl, *b*₃₄ 104°. ¹⁰⁸⁵

AmSCH₂CH₂CH₂Cl, *b*₂₀ 105–20°. ⁸³

PhSCH₂CH₂CH₂Cl, *b*₄ 116–7°, ^{781b} *b*₇ 124–5°, ^{1416b} *b*₁₃ 137°, ^{106b}
*b*₁₅ 142°; ¹¹⁰ *d* 20/4 1.1536, ^{781b} 1.1529; ¹¹⁰ *n* 20/D 1.5752, ^{781b}
 1.57483, ¹¹⁰ *n* 26/D 1.5714. ^{1416b}

p-MeC₆H₄SCH₂CH₂CH₂Cl, *b*₅ 127–8°. ⁸⁸⁶

C₄H₃OCH₂SCH₂CH₂CH₂Cl, *b*₅ 135°; *d* 30/4 1.1637; *n* 25/D
 1.4740. ⁵⁵⁹

EtS(CH₂)₄Cl, *b*₂₀ 97–104°. ¹¹⁰

BuS(CH₂)₄Cl, *b*₁₀ 120–4°. ⁶⁸⁹

PhS(CH₂)₄Cl, *b*₁₂ 155°, ¹¹⁰ *b*₁₄ 159°; *d* 20/4 1.1269; *n* 20/D
 1.56828. ¹¹⁰

p-MeC₆H₄S(CH₂)₄Cl, *b*₃ 148°. ⁸⁸⁶

3-C₄H₃S·S(CH₂)₄Cl, *b*_{1.5} 135°. ⁶⁷⁵

MeS(CH₂)₅Cl, *b*₁₅ 94°; *d* 20/4 1.0300; *n* 20/D 1.48597. ¹¹⁰

EtS(CH₂)₅Cl, *b*₂₅ 122°. ¹¹³

PhS(CH₂)₅Cl, *b*₁ 140°, ¹¹⁰ *b*_{2.5} 114–6°, ^{1416b} *b*₁₄ 174°; *d* 20/4
 1.1065; *n* 20/D 1.58360, ¹¹⁰ 1.5590. ^{1416b}

EtS(CH₂)₆Cl, *b*₂₆ 128–31°. ¹¹³

PhS(CH₂)₆Cl, *m*. 8°. ^{112b}

MeS(CH₂)₇Cl, *b*₈ 100–2°; *d* 20/4 0.9969; *n* 20/D 1.4831. ¹⁰⁷

MeS(CH₂)₈Cl, *b*₃ 113–6°; *d* 20/4 0.9849; *n* 20/D 1.4821. ¹⁰⁷

PhS(CH₂)₈Cl, *m*. 16°. ^{112b}

MeS(CH₂)₉Cl, *b*₂ 118–24°; *d* 20/4 0.9725; *n* 20/D 1.4811. ¹⁰⁷

PhS(CH₂)₉Cl, *m*. 5°. ^{112b}

MeS(CH₂)₁₀Cl, *b*₁ 128–31°; *d* 20/4 0.9641; *n* 20/D 1.4802. ¹⁰⁷

PhS(CH₂)₁₀Cl, *m*. 27.5°. ^{112b}

MeS(CH₂)₁₂Cl, *m*. 4°; *b*₁ 140°; *d* 20/4 0.9497; *n* 20/D 1.4789. ¹⁰⁷

MeS(CH₂)₁₄Cl, *m*. 14°; *b*₁ 155°; *d* 20/4 0.9389; *n* 20/D 1.4780. ¹⁰⁷

MeS(CH₂)₁₆Cl, *m*. 22°; *d* 20/4 0.9294; *n* 20/D 1.4773. ¹⁰⁷

MeS(CH₂)₁₈Cl, *m*. 31°. ¹⁰⁷

4-CH₂:CHSC₅H₆Cl, cyclopentene-2, *b*₇ 65°. ^{202.3}

2-MeSC₆H₁₀Cl, cyclohexane, *b*₁₂ 98–9°, ¹⁵³ *b*₁₅ 105°. ^{202.5}

2-EtSC₆H₁₀Cl, cyclohexane, *b*₁₄ 110°. ^{202.5}

2-PrSC₆H₁₀Cl, cyclohexane, *b*₁₂ 119°. ^{202.5}

2-*i*-PrSC₆H₁₀Cl, cyclohexane, *b*₁₂ 112°. ^{202.5}

2-PhSC₆H₁₀Cl, cyclohexane, $b_{1.5}$ 140–3°. ¹⁴⁷³
 MeSCMe:CHCl, b_{13} 40–1°; d 20/4 1.1157; n 20/D 1.5131. ¹⁵³
 PhSCMe:CHCl, b_{13} 126°; d 20/4 1.1668; n 20/D 1.5898. ¹⁵³
 PhSCCl:CHCl, b_{22} 145–50°. ³³⁴
 MeSCH₂CH:CHCl, b_{21} 55°. ²⁸⁵
 EtSCH₂CH:CHCl, b_{12} 60–1°. ^{1211c}
 Ph₃CSCH₂CHClCH₂Cl, m . 128°. ^{344b}

Chlorinated Methyl Sulfide

(ClCH₂)₂S, m . –37.5°, ¹³⁹⁷ –54°; ⁴⁵¹ b . 156.5°, ⁴⁵¹ 150–5°, ¹¹⁷⁴ 155–6°, ³⁸⁹ 156–6.5°, ¹⁴² b_{747} 156–6.5°, b_{10} 46.2°, ¹³⁹⁷ 47°, ⁴⁵¹ b_{11} 51°, ¹⁴², ^{350a} b_{18} 57.5–8.5°, ^{911c} b_{300} 127.8°; ⁴⁵¹ d 14/4 1.4144, ^{911c} d 20/4 1.4116, ⁴⁵¹ 1.4065, ^{350a} 1.4103; ¹³⁹⁷ n 20/D 1.5275, ³⁸⁹ 1.5313, ⁴⁵¹ 1.5322. ¹³⁹⁷
 ClCH₂SCHCl₂, b_{753} 177.2°, b_5 41.5°, ¹³⁹⁷ b_{29} 85–6°; ⁵⁸⁵ d 20/4 1.5258, ¹³⁹⁷ d 24/4 1.6374; ⁵⁸⁵ n 20/D 1.5395. ¹³⁹⁷
 (Cl₂CH)₂S, b . 189°, ⁴⁵¹ b_{746} 187–8°, b_{10} 62–4°, ¹³⁹⁷ b_{18} 82°, ⁴⁵¹ b_6 72°, b_{48} 103–4°; d 0/4 1.6550, d 20/4 1.6273, ³⁸⁸ 1.6286, ⁴⁵¹ 1.5906; ¹³⁹⁷ n 20/D 1.5464, ³⁸⁸ 1.5426, ⁴⁵¹ 1.5368. ¹³⁹⁷
 ClCH₂SCCl₃, m . –15°; b_{744} 188.7°, b_4 55.4°; d 20/4 1.6222; n 20/D 1.5457. ¹³⁹⁷
 Cl₂CHSCCl₃, m . –36°; b_{751} 203.5°, b_4 57–8°; d 20/4 1.6733; n 20/D 1.5434. ¹³⁹⁷
 (Cl₃C)₂S, m . 10°; ¹³⁹⁷ b . 156–60°, ¹¹⁷⁴ b_{732} 219–20°, $b_{1.5}$ 60.8°; d 20/4 1.7433; n 20/D 1.5560. ¹³⁹⁷
 ClCH₂SCH₂CH₂Cl, b_{750} 193°, b_{10} 77°, ²⁰³ b_{12} 82°, ³⁸⁸ b_{14} 79°; ¹²³⁷ d 0/4 1.359, d 20/4 1.338, ³⁸⁸ d_{20} 1.3553; ²⁰³ n 20/D 1.5311. ³⁸⁸
 ClCH₂SCHMeCl, b_1 40°. ^{202.3}
 Cl₂CHSCH₂CH₂Cl, b_{20} 100°; d 0/4 1.485, d 20/4 1.461; n 20/D 1.5390. ³⁸⁸
 Cl₃CSCH(C₆H₁₃)CH₂Cl, b_2 119–21°. ⁷⁸⁵
 ClCH₂SCHPhCH₂Cl, b_{16} 127–9°. ^{204.5}
 2-ClCH₂SC₆H₁₀Cl, cyclohexane, b_{16} 137–40°. ^{204.5}

Chlorinated Ethyl and Propyl Sulfides

(MeCHCl)₂S, b_{14} 53–5°, ¹⁰⁰⁸ b_{15} 58.5–9.5°, ⁷³ b_{16} 56–7°, ^{911c} b_{18} 59–60.5°, ^{989d} b_{27} 66.5–7.5°, b_{41} 76–7°; d 14/4 1.1992, ^{911c} d 15/4 1.1972. ⁷³
 (ClCH₂CH₂)₂S, m . 14.4°; ⁹⁴⁵, ^{1011b}, ¹⁰¹², ¹¹⁴⁸ b . 217°, ⁶³⁴, ⁹⁴⁵, ^{956b} b_{14}

105°; d 25/4 1.2684; n 30/D 1.5220.¹¹⁴⁸ See the chapter on mustard gas for extensive tables of properties.

$\text{MeCHClSCH}_2\text{CH}_2\text{Cl}$, b_8 68–9°.³⁴⁰

$\text{ClCH}_2\text{CHClSCH}_2\text{CH}_2\text{Cl}$, b_{15} 106.5–8°; ^{911a} d 20/4 1.4038,⁸³⁰ $d_{21.8}$ 1.4219; ^{911a} n 22.5/D 1.5309.⁸³⁰

$\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{CHCl}_2$, $b_{0.05}$ 68–9°; n 20/D 1.5380.⁵²¹

$(\text{ClCH}_2\text{CHCl})_2\text{S}$, b_{15} 115°,^{173, 270} 132–3°,¹⁷ b_{11} 98–108°; ¹⁰⁰⁷ d 20/4 1.634.⁷⁸⁷

$\text{ClCH}_2\text{CH}_2\text{SCHClCHCl}_2$, b_3 118.5–9°;³⁵¹ b_{15} 123–5°; d 15.2/4 1.5441.^{911a}

$\text{ClCH}_2\text{CCl}_2\text{SCHClCHCl}_2$, b_{15} 159.5°,^{1011b} 159–60°; ¹⁰⁹⁷ d 20/4 1.6841,^{1011b, 1097} d 25/4 1.6783;¹⁰⁹⁷ n 20/D 1.5681;^{1011b, 1097} parachor 452.5.^{1011b}

$\text{Cl}_2\text{CHCH}_2\text{SCCl}_2\text{CHCl}_2$, b_{15} 160–1°; d 10.6/4 1.6944.^{911a}

$\text{ClCH}_2\text{CH}_2\text{SCCl}_2\text{CCl}_3$, $b_{4.5}$ 131–2°, b_{15} 159.5–6.0°;³⁵¹ 158–9°;¹⁰⁹⁷ 158.5°; ^{1011b} d 20/4 1.6849,^{1011b, 1097} d 25/4 1.6794;¹⁰⁹⁷ n 20/D 1.5683;^{1011b, 1097} parachor 452.6.^{1011b}

$\text{ClCH}_2\text{CHClSCCl}_2\text{CHCl}_2$, b_{15} 157–9°; d 20/4 1.6825, d 25/4 1.6763; n 20/D 1.5681.¹⁰⁹⁷

$\text{ClCH}_2\text{CHClSCCl}_2\text{CCl}_3$, b_2 132–4°; d 20/4 1.743, d 25/4 1.737; n 20/D 1.5741.¹⁰⁹⁷

$\text{Cl}_2\text{CHCHClSCCl}_2\text{CHCl}_2$, b_{15} 171°,^{1011b} 170–2°;¹⁰⁹⁷ d 20/4 1.7473,^{1011b, 1097} d 25/4 1.7415;¹⁰⁹⁷ n 20/D 1.5471;^{1011b, 1097} parachor 486.1.^{1011b}

$\text{ClCH}_2\text{CH}_2\text{SCHMeCH}_2\text{Cl}$, m. –24.5°; ¹⁴⁶² b_2 69–71°;⁵⁹⁹ b_7 88°, b_{12} 105°;¹⁴⁶² $b_{0.4}$ 53–6°, b_6 89–92°; n 20/D 1.5181, 1.5268.⁵²³

$\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{CHMeCl}(\text{?})$, m. –23°; b_4 88°.¹⁴⁶² Same compound as above?

$(\text{MeCHClCH}_2)_2\text{S}$, m. –40°; ¹⁴⁶² $b_{4.5}$ 95–7°;^{924.5} b_7 94–5°;^{350a} b_{10} 105°;¹⁴⁶² b_{11} 105–6°;¹¹³ b_{13} 107–9.5°;⁵²³ b_{23} 122°;³⁰³ d 25/4 1.1569;^{350a} n 20/D 1.5015–1.5026.⁵²³

$(\text{MeCHClCHMe})_2\text{S}$, b_{11} 121–2°.¹¹¹³

$(\text{ClCH}_2\text{CH}_2\text{CH}_2)_2\text{S}$, b_7 111–2°;^{350a} b_{16} 87°;¹¹⁰ b_{43} 162°; d 20/4 1.175,^{111a} 1.0267,¹¹⁰ d 25/4 1.1774;^{350a} n 20/D 1.5075.^{111a}

$(\text{ClCH}_2\text{CHClCH}_2)_2\text{S}$, b_{15} 181–2°.¹¹¹³

$2\text{-ClC}_5\text{H}_8\text{SCH}_2\text{CH}_2\text{Cl}$, b_{10} 125–7°; d 26/4 1.2370; n 25/D 1.5322.¹¹⁴⁸

$2\text{-ClC}_6\text{H}_{10}\text{SCH}_2\text{CH}_2\text{Cl}$, b_7 139–43°;¹¹⁴⁸ $b_{0.2}$ 84–6°;⁵²² b_1 104–8°;⁵¹⁷ $b_{1.35}$ 115–8°;⁵¹⁸ d 20/4 1.2168;¹¹⁴⁸ n 20/D 1.5490,⁵¹⁷ n 25/D 1.5369.¹¹⁴⁸

Chlorinated Ethyl-Vinyl and Divinyl Sulfides

- $\text{ClCH}_2\text{CHClSCH:CH}_2$, b_{20} $84-5^\circ$.¹⁷
 $\text{ClCH}_2\text{CH}_2\text{SCCl:CH}_2$, b_4 $69.5-70.5^\circ$,⁸³⁰ b_4 92° ; d 20/4 1.3123,⁸⁵¹ 1.3193; n 22.5/D 1.550,⁸³⁰ n 20/D 1.5502.⁸⁵¹
 ClCHMeSCH:CHCl , b_{14} 75° .^{202.3}
 $\text{ClCH}_2\text{CH}_2\text{SCH:CHCl}$, m . -24° ; $b_{0.15}$ 30° , $b_{0.75}$ 46° ,⁵²² $b_{0.3}$ 42° ,¹²³⁷ b_4 $73.5-4.5^\circ$,⁸³⁰ 99° ; d 20/4 1.3103,⁸⁵¹ 1.3280;⁸³⁰ n 20/D 1.5490,⁸⁵¹ 1.5480,⁵²² n 22.5/D 1.5483.⁸³⁰ Two isomers: α , b_2 54° , $b_{6.5}$ $69-73.5^\circ$; d 20/4 1.3031; n 20/D 1.5486; β , b_2 58° , $b_{6.5}$ $79-81^\circ$; d 20/4 1.3220; n 20/D 1.5493.⁹⁸³
 $\text{ClCH}_2\text{CH}_2\text{SCH:CCl}_2$, $b_{3.5}$ $90-1^\circ$.^{351, 830}
 $\text{ClCH}_2\text{CH}_2\text{SCCl:CHCl}$, b_4 $79.7-80.5^\circ$,^{350a} b_{15} $108.5-9^\circ$,³⁵¹ 107° ; ^{1011b, 1097} d 20/4 1.4581,^{350a} 1.4315, d 25/4 1.4255; n 20/D 1.5562; ^{1011b, 1097} parachor 338.4,^{1011a} 338.3.^{1011b}
 $\text{ClCH}_2\text{CH}_2\text{SCCl:CCl}_2$, b_4 $97-8^\circ$, b_{15} $123.5-4.5^\circ$,³⁵¹ 124° ; ^{1011a, 1011b, 1097} d 20/4 1.5425,^{1011b, 1097} 1.537,^{1011a} d 25/4 1.5361,¹⁰⁹⁷ 1.531; ^{1011a} n 20/D 1.5700; ¹⁰⁹⁷ parachor 374.1,^{1011a} 372.3.^{1011b}
 $\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{CH:CHCl}$, b_{17} $113-4^\circ$.²⁸⁵
 $\text{ClCH}_2\text{CHClSCH:CHCl}$, b_{20} $103-4^\circ$.¹⁷
 $\text{ClCH}_2\text{CHClSCCl:CHCl}$, b_{15} $120-1^\circ$; d 20/4 1.5378; d 25/4 1.5315; n 20/D 1.5673.¹⁰⁹⁷
 $\text{ClCH}_2\text{CHClSCCl:CCl}_2$, b_{15} $133-4^\circ$; d 20/4 1.6190; d 25/4 1.6131; n 20/D 1.5770.¹⁰⁹⁷
 $\text{ClCH}_2\text{CCl}_2\text{SCH:CHCl}$, b_{15} 122.5° ; ^{1011b} d 20/4 1.5404,^{1011b, 1097} d 25/4 1.5342; ¹⁰⁹⁷ n 20/D 1.5661; ^{1071b, 1097} parachor 372.8.^{1011b}
 $\text{Cl}_2\text{CHCHClSCCl:CHCl}$, b_{15} 134.5° ; d 20/4 1.6293, ^{1011b, 1097} d 25/4 1.6236; ¹⁰⁹⁷ n 20/D 1.5778; ^{1011b, 1097} parachor 406.2.^{1011b}
 $\text{Cl}_2\text{CHCCl}_2\text{SCCl:CCl}_2$, b_{15} 171° ; d 20/4 1.7473; n 20/D 1.5741; parachor 486.1.^{1011b}
 $\text{CH}_2\text{:CHSCH:CHCl}$, b . $123-4^\circ$.¹⁷
 $(\text{ClCH:CH})_2\text{S}$, b_{12} $71-4^\circ$,⁷⁸⁷ b_{14} $65-75^\circ$,¹⁰⁰⁷ b_{15} $75-80^\circ$; ³³⁴ d 20/4 1.365.⁷⁸⁷
 $(\text{ClCH:CHCH}_2)_2\text{S}$, b_{10} $116-7^\circ$.²⁸⁵

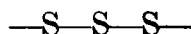
Aromatic Chlorosulfides

- $o\text{-MeSC}_6\text{H}_4\text{Cl}$, b_{15} $116.5-17^\circ$; d 20/4 1.2377; n 20/D 1.6067.^{561, 923}
 $p\text{-MeSC}_6\text{H}_4\text{Cl}$, b . 170° ; d_{25} 1.2224; n 25/D 1.6023.¹⁸⁹

- o -PhSC₆H₄Cl, b_{40} 186°, ¹⁰⁷⁸ b_{11} 163°; dipole moment 2.62.¹¹⁹⁵
 m -PhSC₆H₄Cl, b_{30} 186°, ¹⁰⁷⁸ b_{13} 173–4°; dipole moment 1.89.¹¹⁹⁵
 p -PhSC₆H₄Cl, b . 305–15° with decomposition,⁹⁶¹ b_{10} 167–8°; dipole moment 1.52.¹¹⁹⁵
 p -PhCH₂SC₆H₄Cl, m . 53°. ^{1351b}
 p -MeC₆H₄SC₆H₄Cl- p , m . 73°. ⁸¹⁶
MeSC₆H₃Cl₂-2,4, b_{15} 135–40°; d_{25} 1.3633; n 25/D 1.61678.¹⁸⁹
MeSC₆H₃Cl₂-2,5, m . 51°. ⁵³⁹
MeSC₆Cl₅, m . 96°. ^{1351.5}
EtSC₆Cl₅, m . 44°. ^{1351.5}
(p -ClC₆H₄)₂S, m . 98°, ^{500b} 95°, ¹³²⁹ 93°, ¹⁴⁹ 88°, ⁹⁶¹ 89°; ⁶¹⁵, ^{803b} b_{18} 212°. ¹³²⁹
(2,4-Cl₂C₆H₃)₂S, b_2 197.5–9°. ¹⁰³⁶
(ClC₆H₄CH₂)₂S, *ortho*, m . 52°; ¹³⁰⁸ *para*, m . 42°. ⁷¹⁹, ^{1066a}
(p -ClCPh₂C₆H₄)₂S, m . 163°. ³⁸⁰
 p -ClC₆H₄SCH₂CH₂Cl, m . 34°. ^{66a}
Cl₂C₆H₃SCH₂CH₂Cl, 2,4- b_{15} 218°; 2,5- b_{15} 176°. ^{66a}
2,4,6-Cl₃C₆H₂SCH₂CH₂Cl, m . 71°. ^{66a}
2,4-Cl₂C₆H₃SCHMeCHMeCl, sulfone, *cis* m . 59°; *trans* m . 57.5°. ^{768.5}
 p -MeSC₆H₄CHPhCl, m . 56°. ⁷⁴
 o -MeSC₆H₄CPh₂Cl, m . 126°. ^{192b}
 o -EtSC₆H₄CPh₂Cl, m . 117°. ¹⁹⁸
MeSC₆H₃MeCH₂Cl-4,2, m . 30°; b_6 124–7°. ⁶⁷⁷
MeSC₆H₃(CH₂Cl)₂-2,4, m . 46°. ¹⁴⁷⁹
MeSC₆H₃ClCHPh₂-4,2, m . 98°. ¹⁸⁹
MeSC₆H₃ClCPh₂Cl-4,2, m . 128°. ¹⁸⁹

—S—S—

- 1,3-(MeS)₂C₆H₃Cl-4, b_{18} 177–9°. ¹¹⁰⁷
1,3-(MeS)₂C₆H₂Cl₂-2,5, m . 179°. ⁵³⁹
1,3-(MeS)₂C₆H₂Cl₂-4,6, m . 125°, ¹¹⁰⁸ 123°. ¹⁵¹²
1,3-(ClCH₂S)₂C₆H₂Cl₂-4,6, m . 88.5°. ⁷⁹
(MeSC₆H₄)₂CPhCl, *ortho*, m . 104°; ^{192b} *para*, m . 122°. ^{192b}
(p -MeSC₆H₄)₂CHCCl₃, m . 126°. ²⁴²
(p -MeSC₆H₄)₂C:C:CCl₂, m . 119.5, ²⁴² 119°. ¹⁴¹⁸
(2-MeSC₆H₃Cl-5)₂CHPh, m . 121°. ¹⁸⁹
(2-MeSC₆H₃Cl-5)₂CPhCl, m . 126°. ¹⁸⁹
(2-MeSC₆H₃Cl-5)₂, m . 160°. ¹⁸⁹



- 1,3,5-(MeS)₃C₆H₂Cl-2, m. 94°. ¹¹⁰⁷
 (p-MeSC₆H₄)₃CCl, m. 152°. ^{192b}
 (o-EtSC₆H₄)₃CCl, m. 134°. ¹⁹³
 (2-MeSC₆H₃Cl-5)₃CH, m. 120°. ¹⁸⁹
 (2-MeSC₆H₃Cl-5)₃CCl, m. 170°. ¹⁸⁹

Chloro Multiple Sulfides

- MeSCH₂CH₂SCH₂CH₂Cl, m. 13°; ⁵⁷⁵ b₃ 112°. ²¹¹
 EtSCH₂CH₂SCH₂CH₂Cl, m. 10°. ⁵⁷⁵
 PrSCH₂CH₂SCH₂CH₂Cl, m. 7°. ⁵⁷⁵
 BuSCH₂CH₂SCH₂CH₂Cl, m. -2°. ⁵⁷⁵
 EtSCH₂CHClCH₂SCH₂Ph, b_{0.2} 144-5°. ^{1211a}
 EtSCHClCH₂SEt, b₁₂ 81°. ^{202.5}
 p-MeC₆H₄SCHClCHClSC₆H₄Me-p, m. 138°. ⁵⁰⁷
 PhSCCl:CClSPh, m. 72°. ³³⁴
 ClCH₂CH₂SCH₂SCH₂CH₂Cl, m. 25.5°, ^{350c} 30.7°; ⁵³⁰ b_{0.035} 85°, ^{350c}
 b_{0.03} 80-1°; ⁵³⁰ d 25/4 1.316, d 30/4 1.312. ^{350c}
 (ClCH₂)₂CHSCH₂SCH(CH₂Cl)₂, m. 53°; b₂ 130-2°. ¹⁰⁸²
 ClCH₂CHClCH₂SCH₂SCH₂CHClCH₂Cl, b₃ 145-8°; d 1.35. ¹⁰⁸²
 ClCH₂CH₂SCMe₂SCH₂CH₂Cl, b₂₃ 52-60°. ³⁵⁸
 ClCH₂CH₂SCH₂CH₂SCH₂CH₂Cl, m. 54°, ¹¹⁴, ⁵²⁰ 48°, ⁶⁸⁸ 57°; b₂
 140°. ⁵³⁰
 ClCHMeCH₂SCH₂CH₂SCH₂CHMeCl, m. 15°; b_{0.005} 78-9°. ⁵³⁰
 ClCH₂CH₂SCH₂CH₂SCCl:CCl₂, m. 70.5°. ¹⁰⁹⁷
 ClCH:CHCH₂SCH₂CH₂SCH₂CH:CHCl, b₁₉ 192-4°. ²⁸⁵
 ClCH₂SCHClCHClSCH₂Cl, b₁₂ 42-5°. ²⁰⁵
 ClCH₂CH₂SCHMeCH₂SCH₂CH₂Cl, b_{0.02} 101°; d 25/4 1.231, d
 30/4 1.227. ^{350c}
 ClCH₂CH₂S(CH₂)₃SCH₂CH₂Cl, m. 11.5°, ⁵³⁰ 10.6°; ^{350c} b_{0.04}
 86°, ⁵³⁰ b_{0.055} 97°; d 25/4 1.233, d 30/4 1.229. ^{350c}
 ClCHMeCH₂S(CH₂)₃SCH₂CHMeCl, b_{0.02} 100-8°. ¹⁴⁶²
 ClCH₂CH₂SCHMeCH₂CH₂SCH₂CH₂Cl, b_{0.017} 108°; d 25/4
 1.195, d 30/4 1.191. ^{350c}
 ClCH₂CH₂SCMe₂CH₂CHMeSCH₂CH₂Cl, b_{0.006-0.02} 104-8°; d
 25/4 1.149, d 30/4 1.145. ^{350c}
 ClCH₂CH₂SCH₂CHEtCHPrSCH₂CH₂Cl, b_{0.014} 122°; d 25/4
 1.11. ^{350c}
 ClCH₂CH₂S(CH₂)₄SCH₂CH₂Cl, m. -1°, ⁵³⁰ -0.9°; ^{350c} b_{0.06}

104° ,⁵³⁰ $b_{0.5}$ $160-70^{\circ}$,^{202.7} $b_{0.03-5}$ 113° ; d 25/4 1.200, d 30/4 1.196.^{350c}
 $\text{ClCH}_2\text{CH}_2\text{S}(\text{CH}_2)_5\text{SCH}_2\text{CH}_2\text{Cl}$, m. -6° ,⁵³⁰ -3° ; ^{350c} $b_{0.003}$ 104° ,⁵³⁰ $b_{0.044}$ 128° ; d 25/4 1.173, d 30/4 1.168.^{350c}
 $\text{ClCH}_2\text{CH}_2\text{S}(\text{CH}_2)_6\text{SCH}_2\text{CH}_2\text{Cl}$, m. 15° ,⁵³⁰ 14.4° ; ^{350c} $b_{0.03}$ 112° ,⁵³⁰ $b_{0.055}$ 132° ; d 25/4 1.159, d 30/4 1.155.^{350c}
 $\text{ClCH}_2\text{CH}_2\text{S}(\text{CH}_2)_8\text{SCH}_2\text{CH}_2\text{Cl}$, m. 22° .⁵³⁰
 $\text{ClCH}_2\text{CH}_2\text{S}(\text{CH}_2)_9\text{SCH}_2\text{CH}_2\text{Cl}$, m. 24° .⁵³⁰
 $\text{ClCH}_2\text{CH}_2\text{S}(\text{CH}_2)_{10}\text{SCH}_2\text{CH}_2\text{Cl}$, m. 33° ,^{350c} 32° .⁵³⁰
 $p\text{-ClCH}_2\text{CH}_2\text{SCH}_2\text{C}_6\text{H}_4\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl}$, m. 76.5° .^{350c}
 $\text{S}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl})_2$, m. $73-5^{\circ}$,^{1207, 1462} $72-5^{\circ}$.⁵²⁰
 $\text{S}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CHMeCl})_2$, m. 43.5° .¹⁴⁶²
 $\text{S}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl})_2$, m. 100° .⁵³⁰

Chloro-Hydroxy-Sulfides

$\text{MeSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{Cl}$, $b_{1.4}$ 55° ; d 25/4 1.2250; n 25/D 1.5094.¹³⁹¹
 $\text{EtSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{Cl}$, $b_{1.5}$ 69° ,¹³⁹¹ b_9 100° ; ^{1211a} d 25/4 1.1651; n 25/D 1.5047.¹³⁹¹
 $\text{PrSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{Cl}$, $b_{1.4}$ 80° ; d 25/4 1.1268; n 25/D 1.4986.¹³⁹¹
 $\text{BuSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{Cl}$, $b_{0.5}$ 77° ; d 25/4 1.0883; n 25/D 1.4939.¹³⁹¹
 $\text{AmSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{Cl}$, $b_{0.5}$ 86° ; d 25/4 1.0664; n 25/D 1.4900.¹³⁹¹
 $\text{HexSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{Cl}$, $b_{0.5}$ 97° ; d 25/4 1.0465; n 25/D 1.4880.¹³⁹¹
 $\text{EtSCH}(\text{OH})\text{CCl}_3$, m. 69.5° .¹¹¹⁵
 $\text{C}_{12}\text{H}_{25}\text{SCH}(\text{OH})\text{CCl}_3$, m. 59° .⁴⁸⁷
 $\text{PhSCH}(\text{OH})\text{CCl}_3$, m. 53° .⁹¹
 $\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$, b_2 $152-4^{\circ}$,⁷³ $b_{0.6}$ 100° ,⁵²⁵ $b_{0.5-0.75}$ 87° ; ¹²¹⁷ d 15/4 1.110; ⁷³ n 20/D 1.5188, ⁵²⁵ n 24.5/D 1.5205, ¹²¹⁷ n 27/D 1.5110; Ac., n 25/D 1.4919.¹²⁸⁸
 $\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SH}$, b_{20} $120-7^{\circ}$.³⁵⁸
 $\text{ClCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$, $b_{0.1}$ $93-6^{\circ}$; d 20/4 1.1766; n 20/D 1.5140; Ac., $b_{0.1}$ $86-7^{\circ}$; d 20/4 1.1531; n 20/D 1.4879.⁵⁶³
 $\text{Cl}(\text{CH}_2)_6\text{S}(\text{CH}_2)_6\text{OH}$, $b_{0.25}$ $170-5^{\circ}$; n 21/D 1.4992.¹⁴²⁰
 $\text{Cl}_3\text{CCH}(\text{OH})\text{SCH}(\text{OH})\text{Me}$, m. 97° .⁹⁵⁹
 $(\text{Cl}_3\text{CCHOH})_2\text{S}$, m. 128° ,^{1080, 1487} 127° ; ⁸⁵³ Ac., m. 78° .¹⁴⁸⁷
 $\text{ClCH}:\text{CHCH}_2\text{SCH}_2\text{CH}_2\text{OH}$, b_{16} $135-7.5^{\circ}$.²⁸⁵

- 2,5-Cl₂C₆H₃SCH₂CH₂OH, m. 32°. ^{66a}
 2,4,6-Cl₃C₆H₂SCH₂CH₂OH, m. 41°. ^{66a}
p-ClC₆H₄SCH₂CH(OH)CH₂OH, b₃ 158–62°. ¹⁸³
 MeSC₆H₃ClOH-4,2, b₁₅ 100°; Bz., m. 72°. ¹⁸⁹
 MeSC₆H₃ClCPh₂OH-4,2, m. 112°. ¹⁸⁹
 2,5-HOClC₆H₃SC₆H₄Cl-4, m. 53°. ¹⁰⁹⁴
 (2-HOC₆H₃Cl-5)₂S, m. 174°, ^{538, 1177} 173°; ⁴⁰⁶ Ac., m. 88°. ¹⁰⁹⁴
 (2-HOC₆H₂Cl₂-3,5)₂S, m. 93°. ¹⁰⁹⁴
 (2-HOC₆H₂MeCl-4,5)₂S, m. 178°. ⁹⁴³
 (6-HOC₆H₂MeCl-2,3)₂S, m. 157°. ⁹⁴³
 (2-HOC₆HMe₂Cl-3,6,5)₂S, m. 181°. ⁸⁴⁵
 (2-HOC₆HMe₂Cl-4,6,5)₂S, m. 215°, ⁸⁴⁵ 218°. ⁹⁴³
 (2-HOC₆HMe(C₃H₇)Cl-6,3,5)₂S, m. 111°. ⁸⁴⁵
 β-(1-HOC₁₀H₅Cl-4)₂S, m. 172°. ^{287, 845}
 β-(1-HOC₁₀H₅Cl-4)S(C₁₀H₆OH-2)-α, m. 168°. ⁸⁴⁵
 2(MeSC₆H₃Cl-5)₂CPhOH, m. 135°. ¹⁸⁹
 (2-MeSC₆H₃Cl-5)₃COH, 2 forms, m. 172° and 182°. ¹⁸⁹

Chloro-Ether-Sulfides

- EtO(EtS)CHCHCl₂, b₂₀₋₃₀ 110–5°. ¹⁰⁵¹
 MeOCH₂CH₂SCH₂CH₂Cl, b₁₂ 88°. ⁵⁷⁵
 EtOCH₂CH₂SCH₂CH₂Cl, b₁₁ 92.5°, ⁵⁷⁵ b₂₅ 122°; d₂₀ 1.075. ^{308a}
 PrOCH₂CH₂SCH₂CH₂Cl, b₁₂ 109–10°. ⁵⁷⁵
 BuOCH₂CH₂SCH₂CH₂Cl, b₁₂ 124°. ⁵⁷⁵
 AmSCH₂CH₂OCH₂CH₂Cl, b₁₀ 120–30°. ⁸²
 PhSCH₂CH₂OCH₂CH₂Cl, b₄ 158°; d 15/4 1.1753. ³²⁶
 O(CH₂CH₂SCH₂CH₂Cl)₂, b₂ 174°. ^{1482a}
 O(CH₂CH₂SCH₂CHMeCl)₂, b_{0.02} 95–8°. ¹⁴⁶²
 O(CH₂CH₂SCHMeCH₂Cl)₂, b_{0.01} 90°. ¹⁴⁶²
 O(CH₂CH₂SCH₂CH₂OCH₂CH₂Cl)₂, b_{0.02} 125–30°. ²¹²
 O(CH₂CH₂SCH₂CH₂OCH₂CH₂SCH₂CH₂Cl)₂, m. 28.5°. ²¹²
 O[CH₂CH₂S(CH₂CH₂OCH₂CH₂S)₂CH₂CH₂Cl]₂, m. 37°. ²¹²
 S(CH₂CH₂OCH₂CH₂Cl)₂, b₁ 145°. ²¹²
 S(CH₂CH₂OCH₂CH₂SCH₂CH₂Cl)₂, m. 18°. ²¹²
 S(CH₂CH₂OCH₂CH₂SCH₂CH₂OCH₂CH₂SCH₂CH₂Cl)₂, m.
 33°. ²¹²
 PhSCCl:CHOMe, b₂₀ 160–5°. ³³⁴
p-MeOC₆H₄SCH₂CH₂Cl, b₁ 110°. ^{66a}
 β-C₁₀H₇OCH₂CH₂SCCl:CH₂, m. 57°. ⁸³⁰
 MeSC₆H₃(OMe)Cl-5,2, m. 41°. ^{656c}

$\text{MeSC}_6\text{H}_3(\text{OMe})\text{Cl-5,4}$, m. 36° .^{656c}
 $(2\text{-MeOC}_6\text{H}_3\text{Cl-5})_2\text{S}$, m. 112° .¹¹⁷⁷
 $(2\text{-PrOC}_6\text{H}_3\text{Cl-5})_2\text{S}$, m. 82° .¹⁰⁹⁴
 $p\text{-ClC}_6\text{H}_4\text{SCH}_2\overline{\text{CHCH}_2\text{O}}$, b_3 $132\text{--}7^\circ$.¹⁸³

Bromosulfides

MeSCH_2Br , m. $131\text{--}4^\circ$.¹⁵²
 EtSCH_2Br , b_{45} 67° .¹⁵²
 $\text{CH}_2\text{:CHSCHMeBr}$, b_{12} 50.5° ; d 15/4 1.413.⁷³
 $\text{MeSCH}_2\text{CH}_2\text{Br}$, b_{18} $57\text{--}8^\circ$.¹²³⁷
 $\text{EtSCH}_2\text{CH}_2\text{Br}$, b_8 $57\text{--}8^\circ$,^{350a} b_{12} 60° ,¹²⁷⁷ $55\text{--}7^\circ$,¹²³⁷ b_{29} $83\text{--}6^\circ$; ¹³²³
d 20/4 1.417,¹²⁷⁷ d 25/4 1.3908; ^{350a} n 20/D 1.5242.¹²⁷⁷
 $\text{BuSCH}_2\text{CH}_2\text{Br}$, b_3 74° ; d 0/4 1.2304, d 25/4 1.2054; n 20/D
1.6740.^{1455a}
i-AmSCH₂CH₂Br, b_{13} 102° ; $d_{18.5}$ 1.524.¹²⁷⁷
 $\text{PhSCH}_2\text{CH}_2\text{Br}$, b_{10} $140\text{--}1^\circ$,¹²⁷⁷ b_{11} 135° ,^{21a} b_{13} $132\text{--}6^\circ$; ⁴⁴⁰ d_{18}
1.4456; ¹²⁷⁷ n 25/D 1.6046,^{21a} n_D 1.611.¹²⁷⁷

p-MeC₆H₄SCH₂CH₂Br, b_7 $148\text{--}51^\circ$.⁴⁴⁰
 $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{Br}$, $b_{0.2}$ 111° .¹²³⁷
 $\text{PhCH}_2\text{SCH}_2\text{CHMeBr}$, $b_{0.05}$ $98.5\text{--}99^\circ$.⁷⁹⁵
 $\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{Br}$, b_{18} $155\text{--}60^\circ$,¹⁶⁷ b_{20} $163\text{--}5^\circ$; n 24/D 1.5835.²⁴⁴
 $\text{BrCH}_2\text{SCH}_2\text{Br}$, m. 94° .⁵⁹⁵
 $(\text{MeCHBr})_2\text{S}$, b_{15} 87° ; d 18/4 1.742.⁷³
 MeCHBrSCHClMe , b_{15} 78° ; d 15/4 1.500.⁷³
 $(\text{BrCH}_2\text{CH}_2)_2\text{S}$, m. 34° ,²²⁶ ¹²²⁶, ¹³²³ 31° ; ^{1482a} b. 240° ,¹²²⁶ b_1
 115.5° ,¹²²⁶, ¹³²³ b_2 115° .^{1482a}
 $\text{BrCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{Cl}$, m. 24° ,^{808a} 14° ; $b_{0.3}$ $65\text{--}6^\circ$,¹²³⁷ b_{18} 125--
 32° .^{808a}
 $(\text{BrCH}_2\text{CHBrCH}_2)_2\text{S}$, m. 95.5° .⁸⁹⁴
 $(\text{BrCH:CH})_2\text{S}$, b_{15} $94\text{--}8^\circ$.⁷⁸⁷

Aromatic Bromosulfides

o-MeSC₆H₄Br, m. -24.5° ; ⁶⁷⁶ b. 256° ,¹⁹⁰, ^{192a} b_{13-4} $127\text{--}9^\circ$,¹⁹⁰ b_{35}
 $152\text{--}3^\circ$,⁶⁷⁶ $b_{0.3}$ $81\text{--}3^\circ$; ⁵⁶¹ d_{16} 1.5135,^{192a} d 20/4 1.5216; ¹⁹⁰ n
16/D 1.6340,^{192a} n 20/D 1.6348,⁵⁶¹ 1.6502.¹⁹⁰
p-MeSC₆H₄Br, m. 37.5° ,⁷⁴, ¹⁷⁷, ⁶⁷⁶ 38° ,^{166b}, ^{532c} 35° ,¹⁹⁰ 32° ,^{1351b}
 27° ; ⁶⁷⁰ b. $255\text{--}5.2^\circ$,⁶⁷⁶ $230\text{--}2^\circ$,²³⁵ b_{40} $148.6\text{--}9^\circ$; ⁶⁷⁶ d 39/4
1.4584,¹⁹⁰ d_{41} 1.465; ^{192b} n 39/D 1.61595,¹⁹⁰ n 41/D 1.6149.^{192b}
o-EtSC₆H₄Br, b_{15} 148° ; d_{18} 1.4224; n 18/D 1.60904.¹⁹³

- i*-PrSC₆H₄Br-*o*, b₁₁ 130–5°; d 25/4 1.2767.⁶⁷⁰
i-PrSC₆H₄Br-*p*, b₁₁ 120°; d 25/4 1.2303.⁶⁷⁰
i-BuSC₆H₄Br-*p*, b₁₅ 140–3°; d 25/4 1.1434.⁶⁷⁰
o-PhSC₆H₄Br, b₁₂ 175–7°.⁶⁷⁰
p-PhSC₆H₄Br, m. 25.7°;¹⁷⁶ 177 24.5°; ¹⁴⁹ b₁₄ 187.5°; ¹⁷⁷ b₁₈ 196°.¹⁴⁹
p-MeC₆H₄SC₆H₄Br-*p*, m. 82.5°; b₁₄ 200–5°.¹⁷⁸
m-PhCH₂SC₆H₄Br, m. 41°.¹⁴⁶⁶
p-PhCH₂SC₆H₄Br, m. 65°;^{1351b} 1466 48°.⁶⁷⁰
p-Ph₂CHSC₆H₄Br, m. 107°.¹⁴⁶⁶
 α -C₁₀H₇SC₆H₄Br-*p*, m. 73°; b₁₄ 247°.¹⁷⁸
 β -C₁₀H₇SC₆H₄Br-*p*, m. 114.5°; b₁₄ 253°.¹⁷⁸
ClCH₂CH₂SC₆H₄Br-*p*, m. 39°.^{66a}
Cl₃CSC₆H₄Br-*p*, m. 81.5°.^{1093.5}
MeSC₆H₃MeBr-4,2, m. 15.7°; ⁶⁷⁶ b₂₅ 158°.¹⁵⁰⁸
Cl₃CSC₆H₃MeBr-4,2, m. 57°.¹⁵⁰⁸
MeSC₆H₃Br₂-2,4, m. 49°; b₂₅ 185–6°.⁶⁷⁶
MeSC₆H₂MeBr₂-4,2,5, m. 86°.¹⁵⁰⁸
MeSC₆H₃BrCl-2,4, m. 33°; b₁₅ 140–50°.¹⁸⁹
Cl₃CSC₆H₃BrCl-2,4, m. 69°.^{1093.5}
CH₂:CHCH₂SCH₂C₆H₄Br-*p*, b_{0.5} 110°.⁵⁵

p-ClC₆H₄SC₆H₄Br-*p*, m. 107°.¹⁴⁶⁶
(*m*-BrC₆H₄)₂S, m. 41°.¹⁴⁶⁶
(*p*-BrC₆H₄)₂S, m. 117°;^{500b} 113°;^{1363d} 112.8°;¹⁷⁷ 112.5°;¹²⁰¹
111.5°;¹⁷⁶ 110°;^{803b} 109.5°;⁸⁰⁶ 1486 b₁₁ 225–6°;¹⁷⁶ b₂₀ 243°; b₄₀
268.5°.¹⁷⁷
(*p*-BrC₆H₄CH₂)₂S, m. 59°.⁷¹⁸
MeSC₆H₂MeBrOH-5,3,2, b_{13–4} 162–3°; Ac., m. 52°.¹⁵¹¹
MeSC₆H₂MeBrOH-2,4,3, b_{20–1} 167–9°; Ac., m. 53°.¹⁵⁰³
MeSC₆H₂Br₂OH-2,4,5, m. 84°.^{1506a}
MeSC₆HMeBr₂OH-3,2,5,4, m. 112°.¹⁵⁰³
MeSC₆HMeBr₂OH-3,2,5,6, m. 54°; ¹⁵⁰⁹ 1511 Ac., m. 89°.¹⁵¹¹
MeSC₆HBr₂(CH₂OH)OH-2,5,3,6, m. 126°.¹⁵⁰⁹
MeSC₆HBr₂(CH₂OAc)OH-2,5,3,6, m. 137°; Ac., m. 131°.¹⁵⁰⁹
MeSC₆HBr₂(CH₂Br)OH-2,5,3,6, m. 131°; ¹⁵⁰⁹ 1511 Ac., m.
136°.¹⁵⁰⁹
MeSC₆HBr₂(CH₂OMe)OH-2,5,3,6, m. 82°.¹⁵⁰⁹
MeSC₆H₃Br(OEt)-4,3, m. 17.4°; b. 301–2°; b₃₅ 194–5°; d 17/4
1.4454.⁶⁷⁶
MeSC₆H₂MeBrOMe-2,3,5, m. 64°.¹⁵⁰¹
2-MeSC₁₀H₄Br₂OH-1,5,6, m. 195°; Ac., m. 145°.¹⁵⁰⁵

$(2\text{-HOC}_6\text{H}_3\text{Br-5})_2\text{S}$, m. 180° ,⁴⁰⁶ 176° .^{1362b, 1363a}
 $(2\text{-PrOC}_6\text{H}_3\text{Br-5})_2\text{S}$, m. 84° .¹⁰⁹⁴
 $[2,5,3,6,4\text{-Me}_2\text{Br}_2\text{C}_6(\text{OH})\text{CH}_2]_2\text{S}$, m. 246° .⁵⁰
 $\alpha,\alpha'-(2\text{-HOC}_{10}\text{H}_5\text{Br-6})_2\text{S}$, m. 246° ; diBz., m. 270° .⁸⁴⁵

—S—S—

$1,2\text{-(EtS)}_2\text{C}_6\text{H}_3\text{Br-4}$, m. 54° .⁵⁹²
 $1,3\text{-(MeS)}_2\text{C}_6\text{H}_2\text{Br}_2\text{-4,6}$, m. 142° .¹⁵¹²
 $1,3\text{-(EtS)}_2\text{C}_6\text{H}_2\text{Br}_2\text{-4,6}$, m. 58° .¹⁵¹²
 $1,3\text{-(PhCH}_2\text{S)}_2\text{C}_6\text{H}_2\text{Br}_2\text{-4,6}$, m. 107° .⁴⁶⁸
 $\text{PhCH}_2\text{SCHBrCHBrSCH}_2\text{Ph}$, m. 74° .⁵⁰²
 $p\text{-MeC}_6\text{H}_4\text{SCHBrCHBrSC}_6\text{H}_4\text{Me-}p$, m. 72° .⁵⁰⁷
 $o\text{-O}_2\text{NC}_6\text{H}_4\text{SCHBrCHBrSC}_6\text{H}_4\text{NO}_2\text{-}o$, m. 132° .⁵⁰²
 $p\text{-O}_2\text{NC}_6\text{H}_4\text{SCHBrCHBrSC}_6\text{H}_4\text{NO}_2\text{-}p$, m. 137° .⁵⁰²
 $p\text{-MeC}_6\text{H}_4\text{SCBr:CBrsC}_6\text{H}_4\text{Me-}p$, m. 100° .⁵⁰⁷
 $o\text{-O}_2\text{NC}_6\text{H}_4\text{SCBr:CBrsC}_6\text{H}_4\text{NO}_2\text{-}o$, m. 209° .⁵⁰²

Iodosulfides

MeSCH_2I , b_{36} 72° .¹⁵²
 EtSCH_2I , b_{40} 74° .¹⁵²
 $\text{S}(\text{CH}_2\text{CH}_2\text{I})_2$, m. 62° ,⁵⁸⁶ $55\text{--}60^\circ$,⁶²⁶ $68\text{--}70^\circ$.^{808a}
 $\text{ICH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{I}$, m. 85° .^{808a}
 $\text{S}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{I})_2$, m. 103° .¹⁴⁶²
 $\text{O}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{I})_2$, d 20/4 1.3372.^{1482a}

Aromatic Iodosulfides

$\text{MeSC}_6\text{H}_4\text{I}$, *ortho*, b_{20} 173° ; ¹⁵¹⁹ *meta*, b_{16} 157° ; ¹⁵¹⁴ *para*, m. 45° ,^{532c} 38° .^{1510b}
 $p\text{-Cl}_3\text{CSC}_6\text{H}_4\text{I}$, m. 103° .^{1510b}
 $\text{EtSC}_6\text{H}_4\text{I}$, *ortho*, b_{40} 181° ; d_{10} 1.677; ¹⁹³ *para*, b_{11} $146\text{--}7^\circ$.^{992, 1460}
 $p\text{-PhSC}_6\text{H}_4\text{I}$, m. 35° ; b_{11} 230° .¹⁴⁶⁰
 $\text{MeSC}_6\text{H}_3\text{MeI-2,5}$, b_{16} 176° .¹⁵¹⁶
 $\text{MeSC}_6\text{H}_3\text{MeI-4,2}$, b_{17} $153\text{--}4^\circ$.^{1515b}
 $\text{Cl}_3\text{CSC}_6\text{H}_3\text{MeI-2,5}$, m. 45° .¹⁵¹⁶
 $\text{MeSC}_6\text{H}_3\text{ClI-4,2}$, m. 27° ; b_{15} $175\text{--}85^\circ$; d_{25} 1.881; n 20/D 1.6857.¹⁸⁹
 $\text{MeSC}_6\text{H}_2\text{MeBrI-2,4,5}$, m. 72° .¹⁵¹⁶
 $(p\text{-IC}_6\text{H}_4)_2\text{S}$, m. 139° .^{803b}

Hydroxy-Iodosulfides

- $p\text{-IC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{OH}$, m. 54° .^{66a}
 $(2,5\text{-HOIC}_6\text{H}_3)_2\text{S}$, m. 94° .¹⁰⁹⁴
 $\text{PhSC}_6\text{H}_4\text{I}_2\text{OH-2,6,4-EtOH}$, m. 139° .⁸⁹
 $\text{PhSC}_6\text{H}_4\text{I}_2\text{OH-3,5,4}$, m. 85° .⁸⁹
 $p\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_4\text{I}_2\text{OH-2,6,4}$, m. 154° .⁸⁹
 $p\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_4\text{I}_2\text{OH-3,5,4}$, m. 113° .⁸⁹
 $p\text{-HOC}_6\text{H}_4\text{SC}_6\text{H}_4\text{I-}p$, m. 112° ; b_{2-4} $205\text{--}10^\circ$.⁸⁹
 $p\text{-MeOC}_6\text{H}_4\text{SC}_6\text{H}_4\text{I-}p$, m. 102° ; b_{2-4} 202.5° .⁸⁹

Fluorosulfides

- MeSCF_3 , b_{750} $11.5\text{--}1.7^\circ$.¹³⁹⁷
 $\text{MeSCF}_2\text{CHF}_2$, b. 63° ; d 8/4 1.322; n 8/D 1.3675.⁷⁹⁴
 $\text{EtSCF}_2\text{CHF}_2$, b. 88° ; d 18/4 1.246; n 18/D 1.3735.⁷⁹⁴
 $i\text{-PrSCF}_2\text{CHF}_2$, b. 96° ; d 23/4 1.266; n 20/D 1.3910.⁷⁹⁴
 $\text{PhSCF}_2\text{CHF}_2$, b_{12} 64° ; d 20/4 1.3515; n 20/D 1.4740.⁷⁹⁴
 $\text{MeSCF}_2\text{CHF}_2\text{CF}_3$, b. 87° ; d_{20} 1.380; n 20/D 1.3443.^{794.5}
 $\text{EtSCF}_2\text{CHF}_2\text{CF}_3$, b. $100\text{--}1^\circ$; d_{20} 1.322; n 20/D 1.3548.^{794.5}
 $\text{HOCH}_2\text{CH}_2\text{SCF}_2\text{CHF}_2\text{CF}_3$, b_5 $53\text{--}4^\circ$; d_{20} 1.546.^{794.5}
 F_2CHSCF_3 , b. $0.8\text{--}1.3^\circ$.¹³⁹⁷
 F_3CSCF_3 , b. -22.2° .¹⁹⁵
 $(\text{FCH}_2\text{CH}_2)_2\text{S}$, b_{30} $95\text{--}6^\circ$.⁹⁰⁸
 $(\text{F}_3\text{CCF}_2\text{CF}_2)_2\text{S}(?)$, b_{760} 90° .⁶¹⁶
 $\text{MeS}(\text{CF}_2\text{CF}_2)_{10}\text{SMe}$, m. $113\text{--}6^\circ$.⁶⁰²
 $\text{BuSCH}\cdot\text{CF}_2\text{SBu}$, m. -31.5° ; d 25/4 1.1588; n 25/D 1.4810.¹¹⁴⁶

$$\begin{array}{c} | \\ \text{CF}_2\text{CF}_2 \\ | \\ \text{BuSC}=\text{CSBu} \end{array}$$
, m. -41° ; d 25/4 1.1645; n 25/D 1.4791.¹¹⁴⁶

$$\begin{array}{c} | \\ \text{CF}_2\text{CF}_2 \\ | \\ \beta\text{-C}_{10}\text{H}_7\text{SC}=\text{CSC}_{10}\text{H}_7\text{-}\beta \end{array}$$
, m. 91° .¹¹⁴⁶

$$\begin{array}{c} | \\ \text{CF}_2\text{CF}_2 \end{array}$$

Aromatic Fluorosulfides

- $p\text{-PhSC}_6\text{H}_4\text{F}$, b_{11} $141\text{--}2^\circ$, b_{15} $147\text{--}8^\circ$.⁸⁴²
 $(p\text{-FC}_6\text{H}_4)_2\text{S}$, b_9 $136\text{--}7^\circ$.⁸⁴²
 $o\text{-MeSC}_6\text{H}_4\text{CF}_3$, m. 5.0° ; b_{25} 91° , b_{743} $208\text{--}9^\circ$; d 25/4 1.308; n 25/D 1.509.⁸⁸⁴
 $m\text{-MeSC}_6\text{H}_4\text{CF}_3$, b_{743} $192\text{--}3^\circ$, b_{20} 80° ; d 25/4 1.270; n 20/D 1.497.⁸⁸⁴

- p -MeSC₆H₄CF₃, m. 37°; b_{743} 198.5–9°, b_{12} 88.5°. ⁸⁸⁴
 MeSC₆H₃(CF₃)₂-2,4, m. 21.7°; b_{743} 204–5°, b_{10} 74°; d 25/4 1.453; n 25/D 1.464. ⁸⁸⁴
 MeSC₆H₃(CF₃)₂-2,5, m. 18.5°; b_{743} 195–6°, b_{12} 67°; d 25/4 1.445; n 25/D 1.458. ⁸⁸⁴
 MeSC₆H₃(CF₃)₂-2,6, m. 22.0°; b_{743} 205–6°, b_3 63°; d 25/4 1.453; n 25/D 1.464. ⁸⁸⁴
i-PrSC₆H₂(CF₃)₃-2,4,6, b_{743} 212–3°; d 25/4 1.437; n 25/D 1.428. ⁸⁸⁴
 C₁₂H₂₅SC₆H₂(CF₃)₃-2,4,6, m. 41°. ⁸⁸⁴

Chloro-Fluorosulfides

- MeSCClF₂, m. –100.2°; b_{755} 56.3°; d 20/4 1.298; n 20/D 1.3926. ¹³⁹⁷
 MeSCF₂CHFCl, b. 104°; d 20/4 1.389; n 20/D 1.4083. ⁷⁹⁴
 EtSCF₂CHFCl, b_{100} 69.1°; d 25/4 1.3212; n 25/D 1.4079. ¹¹⁴⁶
 BuSCF₂CHFCl, b_{25} 71.6°; d 25/4 1.224; n 25/D 1.4196. ¹¹⁴⁶
 BuSCF₂CHCl₂, b_1 43°; d 25/4 1.2707; n 25/D 1.4545. ¹¹⁴⁶
 MeSCFClCHFCl, b_{15} 58.5°; d 20/4 1.355; n 20/D 1.5900. ⁷⁹⁴
 EtSCFClCHFCl, b. 138°; d 20/4 1.425; n 20/D 1.4612. ⁷⁹⁴
i-PrSCFClCHFCl, b_{738} 159°; d 25/4 1.302; n 20/D 1.4534. ⁷⁹⁴
 PhSCFClCHFCl, b_3 93°; d 20/4 1.400; n 20/D 1.540. ⁷⁹⁴
 MeSCF:CFCl, b. 88–9°; d 20/4 1.324; n 20/D 1.4390. ⁷⁹⁴
 EtSCF:CFCl, b. 106–7°, b_{736} 120°; d 17/4 1.331, d 20/4 1.282; n 17/D 1.4158, n 20/D 1.4385. ⁷⁹⁴
i-PrSCF:CFCl, b. 135°; d 20/4 1.275; n 20/D 1.4178. ⁷⁹⁴
 PhSCF:CFCl, b. 208°; d 20/4 1.387; n 20/D 1.5030. ⁷⁹⁴
 ClCH₂SCClF₂, b_{740} 105.7°; d 20/4 1.510; n 20/D 1.4408. ¹³⁹⁷
 ClCH₂SCF₃, b_{740} 63.5°; d 20/4 1.4122; n 20/D 1.3818. ¹³⁹⁷
 MeSCHFCFCIOAm, b_3 73°; d 20/4 1.190; n 20/D 1.4640. ⁷⁹⁴
 HOCH₂CH₂SCF₂CHFCl, $b_{0.5}$ 62.5°; d 25/4 1.4793; n 25/D 1.4426. ¹¹⁴⁶

p-Cl₃CSC₆H₄F, b_{18} 122°. ^{1093.5}

Aminosulfides

- EtSCH₂NH₂, C₉H₁₉CO-, m. 38°; C₁₁H₂₃CO- m. 63°; C₁₇H₃₅CO- m. 83.6°. ⁴⁶
 EtSCH₂NHCONHMe, b_1 122.5°. ⁷⁴⁴
 EtSCH₂NHCONHEt, m. 15.5°. ⁷⁴⁴
 (EtSCH₂NH)₂CO, m. 108.5°. ⁷⁴⁴
 EtSCH₂NEt₂, b. 174–5°. ⁸⁹⁵

- $\text{PrSCH}_2\text{NEt}_2$, b. 185° .⁸⁹⁵
 $\text{C}_{12}\text{H}_{25}\text{SCH}_2\text{NH}_2$, b₇ $157-60^\circ$.^{694a}
 $(p\text{-MeC}_6\text{H}_4\text{SCH}_2\text{NH})_2\text{CO}$, m. 126° .^{784.5}
 $(\beta\text{-C}_{10}\text{H}_7\text{SCH}_2\text{NH})_2\text{CO}$, m. 144° .^{784.5}
 $\text{MeSCH}_2\text{CH}_2\text{NH}_2$, b. $146-8^\circ$; HCl, m. 120° ; picrate, m. 119° .¹²³⁹
 $\text{MeSCH}_2\text{CH}_2\text{NEt}_2$, b₁₅₀ $141-5^\circ$.¹⁰⁸⁵
 $\text{EtSCH}_2\text{CH}_2\text{NH}_2$, b. 163° ; HCl, m. 147° .¹²³⁹
 $\text{EtSCH}_2\text{CH}_2\text{NHPh}$, m. 192° .^{732b}
 $\text{EtSCH}_2\text{CH}_2\text{N}(\text{CH}_2)_5$, picrate, m. 70° .^{732b}
 $\text{EtSCH}_2\text{CH}_2\text{NHCH}_2\text{Ph}$, b₁₃ $162-4^\circ$; HCl, m. 215° .^{350a}
 $\text{PrSCH}_2\text{CH}_2\text{NHCH}_2\text{Ph}$, b₁₃ $167-9^\circ$; HCl, m. 175° .^{350a}
 $\text{BuSCH}_2\text{CH}_2\text{NH}_2$, b. 211° ; HCl, m. 118° .²⁰¹
 $\text{BuSCH}_2\text{CH}_2\text{NEt}_2$, b_{2.5} 82° .¹⁰⁸⁵
 $\text{BuSCH}_2\text{CH}_2\text{NHCH}_2\text{Ph}$, b₁₃ $172-230^\circ$; HCl, m. 153° .^{350a}
 $\text{AmSCH}_2\text{CH}_2\text{NH}_2$, b. 231° .²⁰¹
i-AmSCH₂CH₂NH₂, b. 231° ; HCl, m. 167° .²⁰¹
i-AmSCH₂CH₂NHCH₂Ph, b₁₇ $190-250^\circ$; HCl, m. 162° .^{350a}
 $\text{HexSCH}_2\text{CH}_2\text{NH}_2$, b. 252° ; HCl, m. 131° .²⁰¹
 $\text{HexSCH}_2\text{CH}_2\text{N}(\text{CH}_2)_5$, b_{0.9} $150-65^\circ$.¹¹²⁸
 $\text{HepSCH}_2\text{CH}_2\text{NH}_2$, b. 270° ; HCl, m. 121° .²⁰¹
 $\text{OctSCH}_2\text{CH}_2\text{NEt}_2$, b₃ $155-60^\circ$,^{694a} b_{0.7} $116-8^\circ$.¹⁰⁸⁵
 $\text{C}_{16}\text{H}_{33}\text{SCH}_2\text{CH}_2\text{NEt}_2$, b_{0.2} 184° .¹⁰⁸⁵
 $\text{H}_2\text{C}:\text{CHSCH}_2\text{CH}_2\text{NMe}_2$, b. 168.5° .⁸³¹
 $\text{OleylSCH}_2\text{CH}_2\text{NEt}_2$, b_{0.15} $206-9^\circ$.⁸⁴¹
 $\text{PhSCH}_2\text{CH}_2\text{NH}_2\cdot\text{HCl}$, m. 161° .^{527a}
 $\text{PhSCH}_2\text{CH}_2\text{NHMe}$, b₂₈ $149-51^\circ$,¹¹⁵ b₁₅ $136-8^\circ$; HCl, m. 105° .⁴⁴⁰
 $\text{PhSCH}_2\text{CH}_2\text{NMe}_2$, HCl, m. 115° .⁷⁷²
 $\text{PhSCH}_2\text{CH}_2\text{NEt}_2$, b₂₂ $156-8^\circ$,¹¹⁵ b₁₂ $144-8^\circ$; ⁴⁴⁰ HCl, m. 106° .¹¹⁵.
 440
 $\text{PhSCH}_2\text{CH}_2\text{NHCH}_2\text{Ph}$, m. 153° .^{350a}
 $\text{PhSCH}_2\text{CH}_2\text{N}(\text{CH}_2)_5$, b₁₅ $136-8^\circ$; ¹¹²⁸ HCl, m. 186° .⁷⁷²
 $\text{PhSCH}_2\text{CH}_2\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 125° .⁷⁷²
 $\text{PhSCH}_2\text{CH}_2\text{NMePh}$, b₁₄ $222-4^\circ$; picrate, m. 155° .¹⁹⁷
 $p\text{-MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{NEt}_2$, b₁₀ $161-2^\circ$; HCl, m. 108° .⁴⁴⁰
 $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{N}(\text{CH}_2)_5$, b_{1.6} $145-8^\circ$.¹¹²⁸
 $\text{Ph}_2\text{CHSCH}_2\text{CH}_2\text{NMe}_2\cdot\text{HCl}$, m. 183° ,^{1076, 1182} 179° .⁵¹²
 $\text{Ph}_2\text{CHSCH}_2\text{CH}_2\text{N}(\text{CH}_2)_4\cdot\text{HCl}$, m. 166° .¹⁰⁷⁶
 $\text{Ph}_2\text{CHSCH}_2\text{CH}_2\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 182° .¹⁰⁷⁶
 $\text{Ph}_2\text{CHSCH}_2\text{CH}_2\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 180° .¹⁰⁷⁶
 $\text{Ph}(p\text{-MeC}_6\text{H}_4)\text{CHSCH}_2\text{CH}_2\text{NMe}_2\cdot\text{HCl}$, m. 153.2° .⁵¹²

- $\text{PhCH:CHCH}_2\text{SCH}_2\text{CH}_2\text{NEt}_2$, $b_{0.2}$ 150–2°.⁸⁴¹
 $\alpha\text{-C}_{10}\text{H}_7\text{SCH}_2\text{CH}_2\text{NEt}_2$, b_{12} 180–95°; HCl, m. 96°.⁴⁴⁰
 $2\text{-C}_4\text{H}_3\text{S}\cdot\text{SCH}_2\text{CH}_2\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 145.5°.⁶⁷⁵
 $3\text{-C}_4\text{H}_3\text{S}\cdot\text{SCH}_2\text{CH}_2\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 150.5°.⁶⁷⁵
 $3\text{-C}_4\text{H}_3\text{S}\cdot\text{SCH}_2\text{CH}_2\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 110.5°.⁶⁷⁵
 $\text{MeSCHMeCH}_2\text{NH}_2$, b. 158°; picrate, m. 134°.¹⁰²⁰
 $\text{EtSCHMeCH}_2\text{NH}_2$, b. 170–1°; picrate, m. 132°.¹⁰²⁰
 $3\text{-C}_4\text{H}_3\text{S}\cdot\text{SCHMeCH}_2\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 173.5°.⁶⁷⁵
 $3\text{-C}_4\text{H}_3\text{S}\cdot\text{SCHMeCH}_2\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 96°.⁶⁷⁵
 $\text{EtSCH}_2\text{CHMeNHCH}_2\text{Ph}$, b_{11} 157–8°.^{350a}
 $\text{PhSCH}_2\text{CHMeN}(\text{CH}_2)_5\cdot\text{HCl}$, m. 130°.⁷⁷²
 $\text{PhSCH}_2\text{CHMeN}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 148°.⁷⁷²
 $\text{PhSCH}_2\text{CMe}_2\text{NH}_2$, b_4 104–4.5°; $n_{20/D}$ 1.5606.^{946b}
 $\text{PhSCH}_2\text{CHEtNH}_2$, b_8 130–1°; $n_{20/D}$ 1.5647.^{946b}
 $\text{PhCH}_2\text{SCMe}_2\text{CH}_2\text{NH}_2$, b_1 95°; HCl, m. 117°; picrate, m. 207°.³²⁸
 $\text{PhSCHPhCH}_2\text{NH}_2\cdot\text{HCl}$, m. 192°.²⁶³
 $p\text{-MeC}_6\text{H}_4\text{SCHPhCH}_2\text{NH}_2\cdot\text{HCl}$, m. 185°.²⁶³
 $\text{MeSCH}_2\text{CH}_2\text{CH}_2\text{NH}_2$, b. 170°; picrate, m. 127°.¹²³⁹
 $\text{EtSCH}_2\text{CH}_2\text{CH}_2\text{NHCH}_2\text{Ph}$, b_{12} 163–5°; HCl, m. 172°.^{350a}
 $\text{PrSCH}_2\text{CH}_2\text{CH}_2\text{NEt}_2$, b_{38} 138–9°.¹⁰⁸⁵
 $\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{NEt}_2\cdot\text{HCl}$, m. 132°.⁷⁷²
 $\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{NEtPh}$, b_3 191–2°; $n_{21.5/D}$ 1.6072.^{1416b}
 $\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 150°.⁷⁷²
 $\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 143°.⁷⁷²
 $3\text{-C}_4\text{H}_3\text{S}\cdot\text{SCH}_2\text{CH}_2\text{CH}_2\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 121°.⁶⁷⁵
 $2\text{-C}_4\text{H}_3\text{S}\cdot\text{SCH}_2\text{CH}_2\text{CH}_2\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 112°.⁶⁷⁵
 $3\text{-C}_4\text{H}_3\text{S}\cdot\text{SCH}_2\text{CH}_2\text{CH}_2\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 158.5°.⁶⁷⁵
 $\text{PhCH}_2\text{SCHMeCH}_2\text{CH}_2\text{NH}_2$, $b_{0.1}$ 90–2°; $n_{20/D}$ 1.5545; HCl, m. 123°.²⁶⁵
 $\text{PhCH}_2\text{SCMe}_2\text{CH}_2\text{CH}_2\text{NH}_2$, $b_{0.1}$ 102–4°; $n_{20/D}$ 1.5521; HCl, m. 157°.²⁶⁵
 $\text{MeS}(\text{CH}_2)_4\text{NH}_2$, b. 188–90°; HCl, m. 154°; picrate, m. 116–8°.¹²⁴²
 $\text{PhS}(\text{CH}_2)_4\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 138°.⁷⁷²
 $\text{PhS}(\text{CH}_2)_4\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 114°.⁷⁷²
 $3\text{-C}_4\text{H}_3\text{S}\cdot\text{S}(\text{CH}_2)_4\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 133°.⁶⁷⁵
 $3\text{-C}_4\text{H}_3\text{S}\cdot\text{S}(\text{CH}_2)_4\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 150°.⁶⁷⁵
 $\text{PhS}(\text{CH}_2)_5\text{NEtPh}$, b_3 194–5°; $n_{20/D}$ 1.6033.^{1416b}
 $\text{PhS}(\text{CH}_2)_5\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 122°.⁷⁷²

$\text{PhS}(\text{CH}_2)_5\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 120° .⁷⁷²
 $p\text{-HO}_3\text{SC}_6\text{H}_4\text{S}(\text{CH}_2)_5\text{NEtPh}$, m. 125° .^{1416b}
 $3\text{-C}_4\text{H}_3\text{S}\cdot\text{S}(\text{CH}_2)_5\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 91.5° .⁶⁷⁵
 $3\text{-C}_4\text{H}_3\text{S}\cdot\text{S}(\text{CH}_2)_5\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 136.5° .⁶⁷⁵
 $\text{PhS}(\text{CH}_2)_6\text{N}(\text{CH}_2)_5\cdot\text{HCl}$, m. 121° .⁷⁷²
 $\text{PhS}(\text{CH}_2)_6\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}\cdot\text{HCl}$, m. 114° .⁷⁷²
 $(\text{AmSCH}_2\text{CH}_2)_2\text{NH}$, b_{25} 214° .¹⁴³¹
 $(\text{PhSCH}_2\text{CH}_2)_2\text{NC}_{10}\text{H}_7\text{-}\beta$, m. 114° .¹²⁰⁶
 $2\text{-C}_6\text{H}_{11}\text{SC}_6\text{H}_{10}\text{NH}_2$, b_{15} 160° ; ^{1005b}, ¹⁰⁰⁶ HCl , m. 200° .¹⁰⁰⁶

Aromatic Aminosulfides

$o\text{-MeSC}_6\text{H}_4\text{NH}_2$, b. 234° ,¹⁹⁰, ^{192a} b_{15} $133\text{--}4^\circ$; ^{192a} d 17/4 1.1333, d 20/4 1.130; n 17/D 1.62632, n 20/D 1.625.¹⁹⁰
 $m\text{-MeSC}_6\text{H}_4\text{NH}_2$, $b_{0.3}$ $101.5\text{--}2.5^\circ$, b_{11} $144\text{--}6^\circ$,⁵⁶¹ b_{16} $163\text{--}5^\circ$; ¹⁹¹, ¹⁵¹⁴ d 20/4 1.1464; n 20/D 1.6450,¹⁹¹ 1.6427; Ac., m. 78.5° ,⁵⁶¹ 75° .¹⁰⁶⁹, ¹⁵¹⁴
 $p\text{-MeSC}_6\text{H}_4\text{NH}_2$, b_{15} 140° ,^{1510a} $140\text{--}3^\circ$; d 20/4 1.3979,¹⁹⁰ $d_{24.8}$ 1.1390; ^{192b} d 25/4 1.1392; ¹⁴²⁶ n 20/D 1.6395,¹⁹⁰ n 24.3/D 1.6381.^{192b}
 $\text{MeSC}_6\text{H}_4\text{NMe}_2$, *meta*, b_{16} $165\text{--}7^\circ$; ¹⁵¹⁴ *para*, m. 23° .^{1510a}
 $o\text{-EtSC}_6\text{H}_4\text{NH}_2$, b_{15} 144° ; d_{18} 1.087; n 18/D 1.60324.¹⁹³
 $m\text{-EtSC}_6\text{H}_4\text{NH}_2$, Ac., d 25/4 1.0331.¹⁰⁶⁹
 $p\text{-EtSC}_6\text{H}_4\text{NH}_2$, m. 107° ; ^{820.5} b. $280\text{--}1^\circ$; ⁹⁹² d 25/4 1.0978; ¹⁴²⁶ HCl , m. 191° ; ^{820.5}, ⁸²¹ Ac., m. 110° ; Bz., m. 145° .^{49.5}
 $m\text{-BuSC}_6\text{H}_4\text{NH}_2$, Ac., d 25/4 1.0072.¹⁰⁶⁹
 $i\text{-BuSC}_6\text{H}_4\text{NH}_2\text{-}p$, d 25/4 1.0391.¹⁴²⁶
 $p\text{-C}_{18}\text{H}_{37}\text{SC}_6\text{H}_4\text{NH}_2$, m. 71° .¹²⁸⁰
 $o\text{-PhSC}_6\text{H}_4\text{NH}_2$, m. 43° ,^{556a} 42° ,³⁷⁰ 35.5° ; ¹⁷⁹ b_1 $161\text{--}2^\circ$,¹¹²⁹ b_{25} 212° , b_{50} 233° , b_{100} 257.5° .¹⁷⁹
 $p\text{-PhSC}_6\text{H}_4\text{NH}_2$, m. 95.8° ,¹⁴²⁶ 97° ,¹⁵⁰⁰ 96° ,¹⁷⁹, ¹¹²⁹, ¹³⁴⁹ 95° ,^{556a}, ^{649a} 94° ,²⁴ 93° ; ⁷⁵⁴ b_{20} 242.5° , b_{30} 257.5° , b_{100} 282.3° ; ¹⁷⁹ Ac., m. 146° ,⁷⁵⁴ 148° ,^{649a} 144° .¹⁵⁰⁰
 $o\text{-PhCH}_2\text{SC}_6\text{H}_4\text{NH}_2$, m. 45° ,¹²⁸⁷ 42° ; ³⁷⁰ Ac., m. 57° ; Bz., m. 68° .¹²⁸⁷
 $m\text{-PhCH}_2\text{SC}_6\text{H}_4\text{NH}_2$, Ac., m. 74.1° .¹⁰⁶⁹
 $o\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_4\text{NH}_2\text{-}o$, m. 90.5° .^{556a}
 $o\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_4\text{NH}_2\text{-}p$, m. 52° .^{556a}
 $m\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_4\text{NH}_2\text{-}o$, b_1 $174\text{--}7^\circ$; d 20/4 1.157; n 20/D 1.6518.^{556a}
 $m\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_4\text{NH}_2\text{-}p$, m. 73° ; Ac., m. 122° ; formate, m. 73.5° .^{556a}

- p -MeC₆H₄SC₆H₄NH₂-*o*, m. 49°,^{556a} 47°.³⁷⁰
 p -MeC₆H₄SC₆H₄NH₂-*p*, m. 73°,^{556a} 73.5°; HCl, m. 184°.⁸²⁹
 p -*i*-PrC₆H₄SC₆H₄NH₂-*p*, Ac., two forms, m. 94.5° and 109°.^{556a}
 2-C₄H₉S·SC₆H₄NH₂-*o*, HCl, m. 196°.¹³²⁴
 p -Ph₃CSC₆H₄NMe₂, m. 148°.⁸³³
 o -MeSC₆H₄NHCOCH₂Cl, HCl, m. 171°.⁸²¹
 o -EtSC₆H₄NHCOCH₂Cl, HCl, m. 214°.⁸²¹
 p -EtSC₆H₄NHCOCH₂Cl, HCl, m. 125°.⁸²¹
 o -PrSC₆H₄NHCOCH₂Cl, HCl, m. 195° with decomposition.⁸²¹
 o -BuSC₆H₄NHCOCH₂Cl, HCl, m. 185° with decomposition.⁸²¹
 i -AmSC₆H₄NHCOCH₂Cl-*o*, HCl, m. 180° with decomposition.⁸²¹
 MeSC₆H₃MeNH₂-4,2, b₂₀ 130°; Ac., m. 114°.^{1515b}
 MeSC₆H₃MeNH₂-2,4, m. 67°.¹⁰⁹²
 MeSC₆H₃MeNH₂-2,5, HCl, m. 47°; Ac., m. 126°.¹⁵¹⁶
 MeSC₆H₃MeNMe₂-2,5, b₁₇ 159°.¹⁵¹⁶
 EtSC₆H₃MeNH₂-2,4, m. 155°.¹⁰⁹²
 MeSC₆H₃(SO₂NH₂)NH₂-4,2, m. 127°; Ac., m. 160.5°; Bz., m. 190.0°.¹³⁰⁷
 MeSC₆H₃(SO₂NEt₂)NH₂-4,2, m. 112.5°; Ac., m. 118.5°.¹³⁰⁷
 p -BuSCH₂C₆H₄NH₂, d 25/4 1.0292.¹⁴²⁶
 i -AmSCH₂C₆H₄NH₂-*p*, d 25/4 1.0278.¹⁴²⁶
 p -C₁₂H₂₅SCH₂C₆H₄NH₂, m. 40°.¹³²⁵
 p -PhSCH₂C₆H₄NH₂, m. 72°.¹⁴²⁶
 p -MeC₆H₄SCHPhC₆H₄NMe₂-*p*, m. 91°.⁷⁵
 α -MeSC₁₀H₆NH₂-4, m. 54°; Ac., m. 193°.¹⁵¹⁸
 α -MeSC₁₀H₆NMe₂-4, b₁₆₋₇ 199°.¹⁵¹⁸
 β -MeSC₁₀H₆NH₂-6, m. 117°.²³⁷

Bis-Aminosulfides

- (H₂NCH₂CH₂)₂S, b. 231–3°,^{526b} b₂₂ 130–1°,¹⁰²⁸ b₁₇ 118–20°; 84
 2HCl, m. 131°; ^{526b}, ¹⁰²⁸ 2HBr, m. 202°; ⁸⁴ diurea, m. 222°.⁹⁷⁸
 H₂NCH₂CH₂SCH₂CH₂NHSO₂C₆H₄NO₂-*p*, m. 161°.⁸³⁷
 (p -O₂NC₆H₄SO₂NHCH₂CH₂)₂S, m. 185°.⁸³⁷
 (p -H₂NC₆H₄SO₂NHCH₂CH₂)₂S, m. 188.5°; diAc., m. 209°.⁸³⁷
 (Me₂NCH₂CH₂)₂S, b. 168.4°.⁸³¹
 (Et₂NCH₂CH₂)₂S, b₉ 139–40°,⁸³¹ b₅ 160°,¹³⁶⁹ b_{0.2} 88.5°,²⁹⁹ b_{0.3}
 105–6°,⁵⁶² b_{0.4} 64°; ¹⁴ d 20/4 0.8947, 0.8950; ⁵⁶² n 20/D 1.4740,¹⁴
 1.4470, 1.4482,⁵⁶² n 25/D 1.4165; ²⁹⁹ 2HCl, m. 247°,⁸³¹ 247.5°;
 2HBr, m. 237.8°.³¹¹
 (Pr₂NCH₂CH₂)₂S, b₁₉ 194°; d 4/4 0.9007, d 25/4 0.8855; 2HCl,
 m. 164.5°.⁸³¹

- $(\text{Bu}_2\text{NCH}_2\text{CH}_2)_2\text{S}$, b_{10} 205–6°. ⁸³¹
 $[(\cdot\text{CH}_2)_2\text{NCH}_2\text{CH}_2]_2\text{S}$, b_{12} 137–9°. ¹³¹
 $[(\text{CH}_2)_5\text{NCH}_2\text{CH}_2]_2\text{S}$, b_{15} 174.5–6°. ⁸³¹
 $(\text{HO}_2\text{CCH}_2\text{NHCH}_2\text{CH}_2)_2\text{S}$, m. 132°; Et_2 , b_{15} 149–50°. ²⁶²
 $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NMe}_2$, $b_{0.04}$ 53°; n 25/D 1.4660. ^{300a}
 $p\text{-O}_2\text{NC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NMe}_2$, m. 65°; picrate, m. 157°. ^{300a}
 $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NEt}_2$, $b_{1.1}$ 92–4°; n 20/D 1.4912. ²⁹⁹
 $\text{Ph}_2\text{CHCONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NEt}_2$, m. 57°. ²⁹⁶
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NEt}_2$, m. 71°; 2HCl, m. 174°. ^{300a}
 $p\text{-BuNHC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NEt}_2$, dipicrate, m. 149°. ^{300a}
 $\text{EtNHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NEt}_2$, $b_{0.2}$ 88.5°; n 25/D 1.4765. ^{300a}
 $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NBu}_2$, $b_{0.01}$ 89.5°; n 25/D 1.4813. ^{300a}
 $p\text{-O}_2\text{NC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NBu}_2$, dipicrate, m. 80.5°. ^{300a}
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NBu}_2$, dipicrate, m. 95.5°. ^{300a}
 $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{N}(\text{CH}_2)_5$, $b_{0.15}$ 81°; n 25/D 1.5145. ^{300a}
 $p\text{-O}_2\text{NC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{N}(\text{CH}_2)_5$, m. 70.5°; picrate, m. 143°. ^{300a}
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{N}(\text{CH}_2)_5$, m. 72.5°; 2HI, m. 212°. ^{300a}
 $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}$, $b_{0.25}$ 109°; n 25/D 1.5198. ^{300a}
 $p\text{-O}_2\text{NC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}$, m. 102°; picrate, m. 146.5°. ^{300a}
 $p\text{-H}_2\text{NC}_6\text{H}_5\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}$, 2HCl, m. 238°. ^{300a}
 $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NEt}_2$, $b_{0.4}$ 63–5°; n 25/D 1.4882. ^{300a}
 $p\text{-O}_2\text{NC}_6\text{H}_4\text{NHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NEt}_2$, picrate, m. 112.5°. ^{300a}
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{NHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NEt}_2$, m. 105.5°. ^{300a}
 $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{N}(\text{CH}_2)_5$, $b_{0.1}$ 100.5°; n 25/D 1.5118. ^{300a}
 $p\text{-O}_2\text{NC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{N}(\text{CH}_2)_5$, m. 62.5°; picrate, m. 75.5°. ^{300a}
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{N}(\text{CH}_2)_5$, m. 94.8°; 2HI, m. 200°. ^{300a}

- $\text{H}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NEt}_2$, $b_{0.4}$ 80.5°; $n_{20/D}$ 1.4913.³⁰¹
 $p\text{-O}_2\text{NC}_6\text{H}_4\text{CONHCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{NEt}_2$, m. 123.5°.^{300a}
 $(\text{PhCH}_2\text{NHCHMeCH}_2)_2\text{S}$, 2HCl, m. 240°.^{350a}
 $(\text{H}_2\text{NCH}_2\text{CH}_2\text{CH}_2)_2\text{S}$, b. 247–8°.⁸³⁶
 $\text{H}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NEt}_2$, $b_{0.04}$ 86–7°; $n_{20/D}$ 1.4880.^{300a, 827}
 $(\text{H}_2\text{NCH}_2\text{CMe}_2\text{CH}_2)_2\text{S}$, b_2 140°.²⁶⁴
 $(\text{H}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2)_2\text{S}$, b_1 141–3°; picrate, m. 179°; diBz., m. 96°.⁴⁷⁷
 $\text{Me}_2\text{NCH}_2\text{CH}:\text{C}(\text{SPh})\text{CH}_2\text{NMe}_2$, b_8 127–40°.⁴⁹⁰
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{NMe}_2$, 2HCl, m. 224°.^{454a}
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{NEt}_2$, m. 57°; ⁸²¹ HCl, m. 190°.^{454a}
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{CH}_2\text{NEt}_2$, HCl, m. 163°.^{454a}
 $p\text{-ClCH}_2\text{CONHC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{NEt}_2$, m. 77.5°.⁸²¹
 $\text{EtSC}_6\text{H}_4\text{NHCOCH}_2\text{NEt}_2\cdot\text{HCl}$, *ortho*, m. 130°; *para*, m. 123°.⁸²¹
 $o\text{-PrSC}_6\text{H}_4\text{NHCOCH}_2\text{NEt}_2\cdot\text{HCl}$, m. 90°.⁸²¹
 $o\text{-PrSC}_6\text{H}_4\text{NHCOCH}_2\text{NHPr}\cdot\text{HCl}$, m. 132°.⁸²¹
 $p\text{-PrSC}_6\text{H}_4\text{NHCOCH}_2\text{NHBu}\cdot\text{HCl}$, m. 117°.⁸²¹
 $o\text{-BuSC}_6\text{H}_4\text{NHCOCH}_2\text{NEt}_2\cdot\text{HCl}$, m. 90°.⁸²¹
 $o\text{-BuSC}_6\text{H}_4\text{NHCOCH}_2\text{NHBu}\cdot\text{HCl}$, m. 141°.⁸²¹
 $o\text{-i-AmSC}_6\text{H}_4\text{NHCOCH}_2\text{NEt}_2\cdot\text{HCl}$, m. 115°.⁸²¹
 $o\text{-PhSC}_6\text{H}_4\text{NHCH}_2\text{CH}_2\text{NMe}_2$, $b_{1.2}$ 160–2°.^{793b}
 $o\text{-PhSC}_6\text{H}_4\text{NHCH}_2\text{CH}_2\text{NEt}_2$, $b_{0.45}$ 160–1°.^{793b}
 $o\text{-PhSC}_6\text{H}_4\text{NHCH}_2\text{CHMeNMe}_2$, $b_{1.2}$ 177–80°.^{793b}
 $o\text{-PhSC}_6\text{H}_4\text{NHCH}_2\text{CHMeNEt}_2$, m. 64–6°; $b_{1.2}$ 188–9°.^{793b}
 $o\text{-PhSC}_6\text{H}_4\text{NHCH}_2\text{CH}_2\text{CH}_2\text{NMe}_2$, $b_{1.2}$ 172–3°.^{793b}
 $o\text{-PhSC}_6\text{H}_4\text{NHCH}_2\text{CH}_2\text{CH}_2\text{NEt}_2$, $b_{0.7}$ 185–6°.^{793b}

Aromatic Bis-Aminosulfides

- $(o\text{-H}_2\text{NC}_6\text{H}_4)_2\text{S}$, m. 87°,⁹⁴⁰ 86°; ¹⁰⁴¹ diAc., m. 164.5°,⁹⁴⁰ 160°; diBz., m. 163°.¹⁰⁴¹
 $o\text{-H}_2\text{NC}_6\text{H}_4\text{SC}_6\text{H}_4\text{NH}_2\text{-}p$, m. 61°; diAc., m. 208°.⁹⁴⁰
 $(p\text{-H}_2\text{NC}_6\text{H}_4)_2\text{S}$, m. 109°,^{940, 1236} 108°,^{615, 1041, 1426} 106°,^{754, 1223} 105°,⁹⁵² 93°; ⁶⁶³ diAc., m. 216.5°,⁹⁴⁰ 221°,⁶¹⁵ 215°,^{582, 952} 185°,⁶⁶³ 111°; ¹⁰⁴¹ diBz., m. 234°.⁶⁶³
 $(p\text{-Me}_2\text{NC}_6\text{H}_4)_2\text{S}$, m. 127°,⁶¹⁵ 118°.⁹⁵²
 $(2\text{-H}_2\text{NC}_6\text{H}_3\text{Me-5})_2\text{S}$, m. 103.5°,⁹⁵² 103°,^{158, 335b, 1399} also 175°; ^{335b} diAc., m. 165°; ¹⁵⁸ diBz., m. 186°.^{158, 1399}
 $(4\text{-H}_2\text{NC}_6\text{H}_3\text{Me-3})_2\text{S}$, m. 96°; 2HCl, m. 249°; diAc., m. 220°; diBz., m. 233°.^{655.5}

- $p\text{-H}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{C}_6\text{H}_4\text{NH}_2\text{-}p$, m. 93° .¹⁴²⁶
 $p\text{-Me}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{C}_6\text{H}_4\text{NMe}_2\text{-}p$, m. 111.5° .²⁰⁵
 $p\text{-Me}_2\text{NC}_6\text{H}_4\text{SCHMeC}_6\text{H}_4\text{NMe}_2\text{-}p$, m. 125° .^{202.3}
 $(o\text{-H}_2\text{NC}_6\text{H}_4\text{CH}_2)_2\text{S}$, m. 70° .⁷²⁵
 $(p\text{-H}_2\text{NC}_6\text{H}_4\text{CH}_2)_2\text{S}$, m. 105° ,¹⁴²⁶ 104.5° .⁴⁷¹
 $(p\text{-PhC:NC}_6\text{H}_4\text{CH}_2)_2\text{S}$, m. 95° .⁴⁷¹
 $2,4\text{-(H}_2\text{N)}_2\text{C}_6\text{H}_3\text{SC}_6\text{H}_4\text{Me-}m$, m. 112.5° .^{556a}
 $2,4\text{-(H}_2\text{N)}_2\text{C}_6\text{H}_3\text{SC}_6\text{H}_4\text{CHMe}_2\text{-}p$, m. 94.5° .^{556a}
 $2,5\text{-(PhSO}_2\text{NH)}_2\text{C}_6\text{H}_3\text{SBu}$, m. 138.5° .⁶
 $2,5\text{-(PhSO}_2\text{NH)}_2\text{C}_6\text{H}_3\text{SCH}_2\text{CH}_2\text{OH}$, m. $133\text{--}9^\circ$.⁶
 $2,5\text{-(PhSO}_2\text{NH)}_2\text{C}_6\text{H}_3\text{SPh}$, m. 168.5° .⁶
 $2,5\text{-(PhSO}_2\text{NH)}_2\text{C}_6\text{H}_3\text{SCH}_2\text{Ph}$, m. 150.5° .⁶
 $2,5\text{-(PhSO}_2\text{NH)}_2\text{C}_6\text{H}_3\text{SC}_6\text{H}_4\text{Me-}p$, m. 157° .⁶
 $\alpha\text{-(2-HOC}_{10}\text{H}_5\text{-NH}_2\text{-6)}_2\text{S}$, m. 194° ; diBz., m. 177° .⁸
 $p\text{-Et}_2\text{NCH}_2\text{CONHC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{NEt}_2$, HCl, m. 155° .⁸²¹

Bis-Amino Multiple Sulfides



- $\text{H}_2\text{C(SCH}_2\text{CH}_2\text{NH}_2)_2$, 2HCl, m. 18.7° .⁹⁶⁷
 $\text{PrCH(SCH}_2\text{CH}_2\text{NH}_2)_2$, $b_{0.7}$ 151.2° ; HCl, m. 210° ; 2HCl, m. 189° .¹⁰⁸⁵
 $(\text{CH}_2\text{SCH}_2\text{CH}_2\text{NH}_2)_2$, m. 43° ; diAc., m. 138° ; dipicrate, m. 173° .⁴¹²
 $(\text{CH}_2\text{SCH}_2\text{CH}_2\text{NEt}_2)_2$, 2HCl, m. 185.5° .^{1123b}
 $(\text{CH}_2\text{SCH}_2\text{CH}_2\text{NBu}_2)_2$, 2HBr, m. 112.5° .^{1123b}
 $\text{CH}_2(\text{CH}_2\text{SCH}_2\text{CH}_2\text{NH}_2)_2$, $b_{0.4}$ 147° ; diAc., m. 92.5° .^{411a}
 $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NH}_2$, diAc., m. 103° .^{411b}
 $(\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NH}_2)_2$, diAc., m. 122° .^{411a}
 $\text{H}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NH}_2$, $b_{0.6}$ $159\text{--}61^\circ$.^{411b}
 $\text{CH}_2(\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NH}_2)_2$, diAc., m. 93° .^{411a}
 $\text{H}_2\text{C(SC}_6\text{H}_4\text{NH}_2\text{-}p)_2$, m. 100° ,¹¹⁸⁷ 99° ,¹⁴²⁶ 83° ; diAc., m. 193° ,¹²⁶ 212° .¹¹⁸⁷
 $\text{CH}_2(\text{SC}_6\text{H}_4\text{CH}_2\text{NH}_2\text{-}p)_2$, diAc., m. 146° , diBz., m. 165° .¹²⁶
 $\text{PhCH(SC}_6\text{H}_4\text{NH}_2\text{-}p)_2$, m. 131° .¹⁴²⁶
 $(\text{CH}_2\text{SC}_6\text{H}_4\text{NH}_2\text{-}o)_2$, m. 74° ; diAc., m. 195° ; diBz., m. 153° .⁵⁰²
 $(\text{CH}_2\text{SC}_6\text{H}_4\text{NH}_2\text{-}p)_2$, m. 111° .¹⁴²⁶
 $o\text{-H}_2\text{NC}_6\text{H}_4\text{SCH:CHSC}_6\text{H}_4\text{NH}_2\text{-}o$, m. 67° ; 2HCl, m. 201° ; diAc., m. 159° ; diBz., m. 132° .⁵⁰²
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{SCH:CHSC}_6\text{H}_4\text{NH}_2\text{-}p$, diAc., m. 194° .⁵⁰²

(*p*-MeNHCH₂CH₂SC₆H₄-)₂, 2HCl, m. 173°. ¹¹⁵

(*p*-Et₂NCH₂CH₂SC₆H₄-)₂, 2HCl, m. 183°. ¹¹⁵



(*p*-H₂NC₆H₄SCH₂CH₂)₂S, m. 93°. ¹⁴²⁶

(*p*-H₂NC₆H₄SCH₂CH₂)₂SO₂, m. 149°. ¹⁴²⁶

Amino-Hydroxy-Sulfides

Me₂NCH₂CH₂SCH₂CH₂OH, b_{0.05} 70–1°; n 20/D 1.4992. ⁸²⁷

Me₂NCH₂CH₂SCH₂CH₂OCOCHPh₂, citrate, m. 75°. ⁴⁷⁸

Me₂NCH₂CH₂SCH₂CHMeOH, b_{0.05} 64–5°; n 20/D 1.4888; ⁸²⁷

HI, m. 120.8°. ²⁹⁸

Me₂NCH₂CH₂SCH₂CHMeOCOPh, HI, m. 120.8°. ²⁹⁸

Me₂NCH₂CH₂SCH₂CH₂CH₂OH, b_{0.05} 90°; n 20/D 1.4958. ⁸²⁷

Et₂NCH₂CH₂SCH₂CH₂OH, b₂ 104–7°; b_{1.4} 91–2°; n 20/D 1.4940. ³⁰¹

Et₂NCH₂CH₂SCH₂CH₂OCOCHPh₂, citrate, m. 95°. ⁴⁷⁸

Et₂NCH₂CH₂SCH₂CHMeOH, b_{0.15} 92–4°; n 20/D 1.4867. ⁸²⁷

Et₂NCH₂CH₂SCH₂CH(CH:CH₂)OH, b_{1.5} 111°. ⁵¹³

Et₂NCH₂CH₂SCH₂CHPhOH, b_{0.5} 132°; d 20/4 1.0175; n 25/D 1.5423. ⁵⁵⁸

Et₂NCH₂CH₂SCH₂CH₂CH₂OH, b₁₋₂ 124–6°, ⁵⁶⁴ b_{0.4} 90.5°; n 20/D 1.4955. ³⁰¹

Et₂NCH₂CH₂SCH₂CH₂CH₂OCOCHPh₂, citrate, m. 113°. ⁴⁷⁸

Et₂NCH₂CH₂CH₂SCH₂CH₂OH, b_{0.1} 100–2°, ⁵⁶³ b_{0.7} 106–7°, ⁸²⁷ b_{1.1} 105°; d 20/4 0.9830; n 20/D 1.4957, ^{556b} 1.4945. ⁸²⁷

Et₂NCH₂CH₂CH₂SCH₂CHMeOH, b_{0.03} 82–3°; n 20/D 1.4871. ⁸²⁷

Et₂NCH₂CH₂CH₂SCH₂CH(CH:CH₂)OH, b_{0.8} 123°. ⁵¹³

Et₂NCH₂CH₂CH₂SCH₂CHPhOH, b_{0.5} 132°, ⁵¹³ 149–52°; d 20/4 1.0104; n 20/D 1.5363. ⁵⁵⁸

Et₂NCH₂CH₂CH₂SCH₂CH₂CH₂OH, b_{0.1} 126–9°, ⁵⁶³ b_{0.2} 108–10°; n 20/D 1.4928. ⁸²⁷

Et₂NCH₂CH₂SCH₂CH₂CH₂CH₂OH, b_{0.03} 109–10°; n 20/D 1.4942. ⁸²⁷

Et₂NCH₂CH₂CH₂SCH₂CH₂CH₂CH₂OH, b_{0.4} 109–10°; n 20/D 1.4919. ⁸²⁷

(CH₂)₅NCH₂CH₂CH₂SCH₂CH₂OH, b_{0.1} 113–5°; n 20/D 1.5188. ⁸²⁷

(CH₂)₅NCH₂CH₂CH₂SCH₂CHMeOH, b_{0.05} 104–5°; n 20/D 1.5098. ⁸²⁷

- $(\text{CH}_2)_5\text{NCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{OH}$, $b_{0.01}$ 115–20°; n 20/D 1.5148.⁸²⁷
 $\text{Et}_2\text{NCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OH}$, $b_{0.2}$ 120°; n 20/D 1.5268.³⁰¹
 $\text{Et}_2\text{NCH}_2\text{CH}(\text{OH})\text{CH}_2\text{SC}_{12}\text{H}_{25}$, $b_{0.2}$ 151–2°; d 20/4 0.9033; n 20/D 1.4739.⁹²⁶
 $\text{Et}_2\text{NCH}_2\text{CH}(\text{OH})\text{CH}_2\text{SPh}$, $b_{0.8}$ 125–8°.⁵¹³
 $\text{Et}_2\text{NCH}_2\text{CH}(\text{OH})\text{CH}_2\text{SCHPh}_2$, $b_{0.2}$ 189–92°.¹⁶⁹
 $\text{Et}_2\text{NCH}_2\text{CH}(\text{OH})\text{CH}_2\text{SC}_6\text{H}_4\text{Me-}p$, $b_{0.8}$ 137°.⁵¹³
 $\text{Et}_2\text{NCH}_2\text{CH}(\text{OH})\text{CH}_2\text{SC}_6\text{H}_4\text{Cl-}p$, $b_{1.5}$ 149–51°.⁵¹³
 $\text{Bu}_2\text{NCH}_2\text{CH}(\text{OH})\text{SC}_6\text{H}_4\text{Cl-}p$, HCl , $m.$ 91°.¹⁸³
 $\text{MeSCH}_2\text{CH}_2\text{CH}(\text{NH}_2)\text{CPh}_2\text{OH}$, $m.$ 104.5°.⁷⁴⁵
 $o\text{-HOCH}_2\text{CH}_2\text{SC}_6\text{H}_4\text{NHCOPh}$, $m.$ 75°; Bz. , $m.$ 84°.⁵¹⁵
 $p\text{-HOCH}_2\text{CH}_2\text{SC}_6\text{H}_4\text{NH}_2$, b_{15} 190°.¹³³⁹
 $o\text{-HOCHMeCH}_2\text{SC}_6\text{H}_4\text{NH}_2$, $b_{1.5}$ 140°.⁵¹⁵
 $o\text{-HOCHPhCH}_2\text{SC}_6\text{H}_4\text{NH}_2$, $m.$ 58°; $b_{1.5}$ 192°; HCl , $m.$ 172°.⁵¹⁵
 $p\text{-HOCHPhCH}_2\text{SC}_6\text{H}_4\text{NH}_2$, $b_{0.001}$ 185°.⁵¹³
 $p\text{-HOCH}(\text{CH}:\text{CH}_2)\text{CH}_2\text{SC}_6\text{H}_4\text{NH}_2$, $b_{0.8}$ 165–8°.⁵¹³
 $p\text{-HOC}_6\text{H}_4\text{SC}_6\text{H}_4\text{NH}_2\text{-}m$, $m.$ 84.5°.⁴⁸³
 $p\text{-HOC}_6\text{H}_4\text{SC}_6\text{H}_4\text{NH}_2\text{-}p$, $m.$ 152°; Ac. , $m.$ 159°.⁴⁸²
 $2,4\text{-(HO)}_2\text{C}_6\text{H}_3\text{SC}_6\text{H}_4\text{NH}_2\text{-}p$, $m.$ 152°; Ac. , $m.$ 158.5°.⁴⁸²
 $2,4\text{-Me(HO)C}_6\text{H}_3\text{SC}_6\text{H}_4\text{NH}_2\text{-}p$, $m.$ 150°; Ac. , $m.$ 128.5°.⁴⁸²
 $5,2,4\text{-Me(Me}_2\text{CH)(HO)C}_6\text{H}_2\text{SC}_6\text{H}_4\text{NH}_2\text{-}p$, $m.$ 113°; Ac. , $m.$ 91°.⁴⁸²
 $\alpha\text{-(}\beta\text{-HOC}_{10}\text{H}_7\text{)SC}_6\text{H}_4\text{NH}_2\text{-}m$, $m.$ 193°; diAc. , $m.$ 164°.⁴⁸³
 $1\text{-HOCH}_2\text{SC}_{10}\text{H}_8\text{NH}_2\text{-}4$, diAc. , $m.$ 160°.¹⁵¹⁸

—N—N—

- $\text{Et}_2\text{NCH}_2\text{CH}(\text{OH})\text{CH}_2\text{SCH}_2\text{CH}_2\text{NEt}_2$, $b_{0.8}$ 121–2°.⁵¹³
 $\text{Et}_2\text{NCH}_2\text{CH}(\text{OH})\text{CH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NEt}_2$, $b_{0.5}$ 131–4°.⁵¹³
 $\text{Et}_2\text{NCH}_2\text{CH}(\text{OH})\text{CH}_2\text{SC}_6\text{H}_4\text{NH}_2\text{-}p$, b_1 153°.⁵¹³
 $\text{Et}_2\text{NCH}_2\text{CH}(\text{OH})\text{CH}_2\text{SC}_6\text{H}_4\text{NMe}_2\text{-}p$, $b_{0.001}$ 145–7°.⁵¹³
 $\alpha\text{-(2-HOC}_{10}\text{H}_5\text{NH}_2\text{-6)}_2\text{S}$, $m.$ 194°; diBz. , $m.$ 177°.⁸
 $(\text{MeSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{NHCH}_2\cdot)_2$, $m.$ 123°.¹³⁹²
 $(\text{EtSCH}_2\text{CH}(\text{OH})\text{CH}_2\text{NHCH}_2\cdot)_2$, $m.$ 119°.¹³⁹²

Amino-Ether-Sulfides

- $p\text{-PhSC}_6\text{H}_4\text{OCH}_2\text{CH}_2\text{NMe}_2$, HCl , $m.$ 186°.¹¹²⁹
 $o\text{-PhSC}_6\text{H}_4\text{OCH}_2\text{CH}_2\text{NEt}_2$, b_1 150–5°; HCl , $m.$ 106°; MeI , $m.$ 139°.¹¹²⁹

p -PhSC₆H₄OCH₂CH₂NEt₂, HCl, m. 125°. ¹¹²⁹
 PhSCH(C₆H₆OMe- p)CH₂NH₂, HCl, m. 178°. ²⁶³
 p -MeC₆H₄SCH(C₆H₄OMe- p)CH₂NH₂, HCl, m. 197°. ²⁶³
 PhSCH(C₆H₃O₂CH₂-3,4)CH₂NH₂, HCl, m. 187°. ²⁶³
 p -MeOC₆H₄SC₆H₄NH₂- p , m. 96°; HCl, m. 207°. ⁸⁹
 p -EtOC₆H₄SC₆H₄NH₂- p , m. 53°, ⁸²⁹ 51°; ¹⁴⁴ HCl, m. 185°; ⁸²⁹
 Ac., m. 129°. ¹⁴⁴
 H₂NCH₂CH₂OCH₂CH₂SCH₂CH₂NH₂, b_{1.5} 120–1°. ^{411a}

Amino-Halo-Sulfides

ClCH₂CH₂SCH₂CH₂NH₂, HCl, m. 78°; picrate, m. 105°. ^{527b}
 ClCH₂CH₂SCH₂CH₂NEt₂, m. 85°. ³⁰¹
 ClCH₂CH₂CH₂SCH₂CH₂NEt₂, b₁ 90–2°, b_{0.3} 74°, ³⁰¹ b₂ 100–2°; ⁵⁶⁴
 n 20/D 1.4886. ³⁰¹
 ClCH₂CH₂SCH₂CH₂CH₂NEt₂, b_{0.1} 71–5°, ⁵⁶³ b_{0.3} 84–95°; d 20/4
 1.000; n 20/D 1.4890. ^{556b}
 ClCH₂CH₂CH₂SCH₂CH₂CH₂NEt₂, b_{0.1} 95–7°, ⁵⁶³ b_{0.025} 80–2°; ⁸²⁷
 d 20/4 0.9980; n 20/D 1.4890, ⁵⁶³ 1.4875. ⁸²⁷
 ClCH₂CH₂SCH₂CH₂SCH₂CH₂NEt₂, b₁ 130°; n 20/D 1.5245. ³⁰¹
 ClCH:CCISCH₂CH₂NHCH₂Ph·HCl, m. 176°. ^{350a}
 MeSC₆H₃ClNH₂-5,2, Ac., m. 110°; Bz., m. 106°. ^{656b}
 MeSC₆H₄ClNH₂-4,2, m. 29°; b. 273–4°, b₁₅ 165–7°; d 25/4
 1.2717; n 25/D 1.63118; HCl, m. 199°; Ac., m. 96°. ¹⁸⁹
 MeSC₆H₃ClNH₂-3,4, HCl, m. 218°; Ac., m. 129°. ^{656b}
 EtSC₆H₃ClNH₂-4,2, b₂₀ 164–5°; Ac., m. 69°; Bz., m. 77°. ¹³⁰⁷
 PhCH₂SC₆H₃ClNH₂-4,2, m. 57°, ¹¹⁷⁸ 54°; Ac., m. 88°. ¹³⁰⁷
 PhCH₂SC₆H₃ClNH₂-6,2, b₁₅ 223°; Ac., m. 102°. ¹³⁰⁷
 Cl₃CSC₆H₂Cl₂NH₂-2,4,5, Ac., m. 160°. ¹⁵¹⁴
 ClC₆H₄SC₆H₄NH₂- p , *ortho*, m. 78°; ^{556a} *meta*, m. 72.5°; *para*, m.
 79°. ^{556a}
 5,2-Cl(HO)₆H₃SC₆H₄NH₂- p , m. 115°. ¹⁰⁹⁴
 ClC₆H₄SC₆H₃(NH₂)₂-2,4, *meta*, m. 95°; ^{556a} *para*, m. 142°. ^{556a}
 2,4-H₂NCIC₆H₃SC₆H₄NMe₂- p , m. 146°. ¹¹⁷¹
 (2-H₂NC₆H₂Cl₂-4,6)₂S, m. 147.5°. ¹³⁰⁷
 p -ClC₆H₄CHPhSCH₂CH₂NMe₂, HCl, m. 147°. ⁵¹²
 (p -ClC₆H₄)₂CHSCH₂CH₂NMe₂, HCl, m. 181.5°. ⁵¹²
 p -BrC₆H₄SC₆H₄NH₂- p , m. 76°. ¹⁴⁶⁶
 EtSC₆H₃BrNH₂-4,2, b₁₅ 169.5–70°; Bz., m. 83.5°. ¹³⁰⁷
 PhCH₂SC₆H₃BrNH₂-4,2, m. 55.5°; Bz., m. 69°. ¹³⁰⁷

$\text{MeSC}_6\text{H}_2\text{MeBrNH}_2$ -2,4,5, m. 73° .^{151b}
 $p\text{-Me}_2\text{NC}_6\text{H}_4\text{SC}_6\text{H}_3\text{BrNH}_2$ -4,2, m. 132° .¹¹⁷¹
 $\text{PhSC}_6\text{H}_2\text{I}_2\text{NH}_2$ -2,6,4, m. 146° ; HCl, m. 192° .⁸⁹
 $p\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_2\text{I}_2\text{NH}_2$ -2,6,4, m. 198° ; HCl, m. 182° .⁸⁹
 $p\text{-MeOC}_6\text{H}_4\text{SC}_6\text{H}_2\text{I}_2\text{NH}_2$ -2,6,4, m. 173° ; HCl, m. 230° .¹²²⁴
 $p\text{-EtOC}_6\text{H}_4\text{SC}_6\text{H}_2\text{I}_2\text{NH}_2$ -2,6,4, m. 152° ; Ac., m. 237° .¹⁴⁴
 $(2\text{-H}_2\text{NC}_6\text{H}_3\text{I-4})_2\text{S}$, m. 155.5° .^{96b}

Nitrogen Bases

PIPERIDINE

$2\text{-MeSC}_5\text{H}_8\text{N}$, tetrahydropyridine, b_{28} $91\text{--}2^\circ$; d 22/4 1.044; n 22/D 1.530; HI, m. 175° .¹¹⁶²
 $2\text{-PhCH}_2\text{SC}_5\text{H}_8\text{N}$, tetrahydropyridine, b_7 $159\text{--}60^\circ$; HBr, m. 172° .¹¹⁶²
 $2\text{-C}_5\text{H}_4\text{NSC}_5\text{H}_8\text{N-2}$, tetrahydropyridine, HBr, m. 205° .¹¹⁶²
 $4,4\text{-dipiperidyl-sulfide-N,N'-dioxide}$, m. 230° .^{713.5}

PYRIDINE

$2\text{-MeSC}_5\text{H}_4\text{N}$, b_{22} 91° ; d 22/4 1.120; n 22/D 1.589.¹¹⁶²
 $2\text{-EtSC}_5\text{H}_4\text{N}$, b_{15} 89.5° ; d 22/4 1.073; n 22/D 1.569; HI, m. 103° .¹¹⁶²
 $2\text{-PrSC}_5\text{H}_4\text{N}$, b_1 $53\text{--}5^\circ$; d 20/4 1.0466; n 20/D 1.5595.¹³⁶⁰
 $2\text{-BuSC}_5\text{H}_4\text{N}$, b_{13} 115° ; d 22/4 1.024; n 22/D 1.549; HI, m. 132° .¹¹⁶²
 $2\text{-CH}_2\text{:CHCH}_2\text{SC}_5\text{H}_4\text{N}$, b_3 $85\text{--}7^\circ$; d 20/4 1.0778; n 20/D 1.5811.¹³⁶⁰
 $2\text{-MeCH:CHSC}_5\text{H}_4\text{N}$, $b_{0.6}$ 72° ; d 20/4 1.0842; n 20/D 1.5970.¹³⁶⁰
 $2\text{-PhCH}_2\text{SC}_5\text{H}_4\text{N}$, b_4 $153\text{--}4^\circ$; d 22/4 1.145; n 22/D 1.628; HBr, m. 136° .¹¹⁶²
 $2\text{-MeSCH}_2\text{CH}_2\text{C}_5\text{H}_4\text{N}$, $b_{1.5}$ 85.5° ; d_{25} 1.0537; n 25/D 1.5492.^{1416c}
 $2\text{-BuSCH}_2\text{CH}_2\text{C}_5\text{H}_4\text{N}$, b_3 $108\text{--}22^\circ$; d_{24} 0.9970; n 25/D 1.5268.^{1416c}
 $2\text{-C}_{12}\text{H}_{25}\text{SCH}_2\text{CH}_2\text{C}_5\text{H}_4\text{N}$, $b_{0.12}$ 182° ; d_{25} 0.9634; n 25/D 1.5038.^{1416c}
 $2\text{-PhCH}_2\text{SCH}_2\text{CH}_2\text{C}_5\text{H}_4\text{N}$, b_1 146° ; d_{25} 1.0976; n 25/D 1.5920.^{1416c}
 $2\text{-p-MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{C}_5\text{H}_4\text{N}$, $b_{0.7}$ $152\text{--}3^\circ$; d_{25} 1.0925; n 25/D 1.5962.^{1416c}

- 2- β -C₁₀H₇SCH₂CH₂C₅H₄N, b_{0.05} 168–70°; d₂₅ 1.1422; n_{25/D} 1.6556.^{1416c}
 (2-C₅H₄N)₂S, b₉ 172–3°; d_{22/4} 1.227; n_{22/D} 1.648; picrate, m. 120°.¹¹⁶²
 (4-C₅H₄NO)₂S, m. 228–30°.^{713.5}
 2-MeSC₅H₂NI₂-3,5, m. 66°.⁷⁹¹
 2-MeSC₅H₃N·NO₂-5, m. 113°.¹³⁵³
 2-EtSC₅H₃N·NO₂-5, m. 63°.¹³⁵³
 2-*i*-PrSC₅H₃N·NO₂-5, b. 270–2°.¹³⁵³
 2-BuSC₅H₃N·NO₂-5, b₂₀ 185–90°.¹³⁵⁵
 2-*i*-BuSC₅H₃N·NO₂-5, b. 270°.¹³⁵³
 [2-(5-O₂N·NC₅H₃)]₂S, m. 137°.¹³⁵³
 2-MeSC₅H₃N·NH₂-5, m. 195°.¹³⁵³
 2-EtSC₅H₃N·NH₂-5, m. 172°.¹³⁵³
 2-*i*-PrSC₅H₃N·NH₂-5, HCl, m. 158°.¹³⁵³
 2-BuSC₅H₃N·NH₂-5, b₂₅ 190–5°.¹³⁵⁵
 2-*i*-BuSC₅H₃N·NH₂-5, HCl, m. 153°.¹³⁵³
 2-MeSC₅H₂N(NO₂)NH₂-3,5, m. 170°.¹³⁵⁵
 2-EtSC₅H₂N(NO₂)NH₂-3,5, m. 95°.¹³⁵⁵
 2-BuSC₅H₂N(NO₂)NH₂-3,5, m. 142°.¹³⁵⁵
 4-O₂NC₆H₄S(4-C₅H₇NO), m. 155°.^{713.5}

QUINOLINE

- 8-MeSC₉H₆N, m. 80°.¹³⁶⁸
 2-PhSC₉H₆N, m. 49°; b_{0.6} 164–6°.⁷⁰³
 2-(*p*-MeSC₆H₄S)C₉H₆N, m. 68°.⁵⁵⁵
 2-(MeSC₆H₄)C₉H₅NEt-3, m. 81°; b₈ 22°; picrate, m. 191°.²⁴³
 2-(MeSC₆H₄)C₉H₄NMeEt-6,3, m. 98°; b₅ 225°; picrate, m. 190°.²⁴³
 4-BuSC₉H₄NMeOMe-2,6, m. 68°.¹¹⁶³
 4-C₁₄H₂₉SC₉H₄NMeOMe-2,6, m. 64.5°.¹¹⁶³
 6-MeSC₉H₅N·NO₂-5, m. 139°.⁹²⁶
 6-C₁₂H₂₅SC₉H₅N·NO₂-5, m. 45°.⁹²⁶
 6-C₁₂H₂₅SC₉H₅N·NH₂-5, m. 60°; 2HCl, m. 157°; Ac., m. 122°.⁹²⁶
 5-(*p*-MeC₆H₄S)C₉H₅NOH-6, m. 138°.²⁰⁸
 5,7-(*p*-MeC₆H₄S)₂C₉H₄NOH-8, m. 126°.²⁰⁸
 5,7-(2',5'-Cl₂C₆H₂S)C₉H₄NOH-8, m. 196°.²⁰⁸
 4-O₂NC₆H₄SC₉H₆NO-4, m. 212°.^{713.5}
 (4-C₉H₆N)₂S, m. 231°.^{713.5}

Quaternary Salts

TABLE 4.7

Quaternary Salts

Iodides

R	Me ₃ NCH ₂ SR·I ¹¹⁶⁵ M.p. (°C)	Et ₃ NCH ₂ SR·I ¹¹⁶⁵ M.p. (°C)
Methyl	137°	134–6°
Ethyl	120°	103.5°
Propyl	111–3°	81–5°
<i>i</i> -Propyl	143–5°	133°
Butyl	123–6°	—
<i>i</i> -Butyl	154°	101°

CHLORIDES

C₁₂H₂₅SCH₂NMe₃Cl, m. 193°, ⁶⁹⁹ 180°. ¹³⁶⁶
 C₁₂H₂₅SCH₂N(C₁₂H₂₅)Me₂Cl, m. 165°, ¹³⁶⁶ 164°. ⁶⁹⁹
 MeSCH₂CH₂N(C₁₂H₂₅)Me₂Cl, m. 50°. ¹³³⁸
 EtSCH₂CH₂N(C₁₂H₂₅)Me₂Cl, m. 172°. ¹⁴⁹⁰
 EtSCH₂CH₂N(C₁₆H₃₃)Me₂Cl, m. 163°. ¹⁴⁹⁰
 PhSCH₂CH₂N(C₁₂H₂₅)Me₂Cl, m. 82.5°. ¹³³⁸
 PhSCH₂CH₂N(C₁₆H₃₃)Me₂Cl, m. 154°. ¹⁴⁹⁰
 PhCH₂SCH₂CH₂N(C₁₂H₂₅)Me₂Cl, m. 191°. ¹⁴⁹⁰
 PhCH₂SCH₂CH₂N(C₁₆H₃₃)Me₂Cl, m. 115°. ¹⁴⁹⁰
p-BrC₆H₄SCH₂CH₂N(C₁₂H₂₅)Me₂Cl, m. 181°. ¹⁴⁹⁰
p-BrC₆H₄SCH₂CH₂N(C₁₆H₃₃)Me₂Cl, m. 115°. ¹⁴⁹⁰
o-BrC₆H₄CH₂SCH₂CH₂N(C₁₂H₂₅)Me₂Cl, m. 117°. ¹⁴⁹⁰
o-BrC₆H₄CH₂SCH₂CH₂N(C₁₆H₃₃)Me₂Cl, m. 153°. ¹⁴⁹⁰
p-BrC₆H₄CH₂SCH₂CH₂N(C₁₂H₂₅)Me₂Cl, m. 99°. ¹⁴⁹⁰
p-BrC₆H₄CH₂SCH₂CH₂N(C₁₆H₃₃)Me₂Cl, m. 158°. ¹⁴⁹⁰
 S[CH₂CH₂N(C₁₂H₂₅)Me₂]₂Cl₂, m. 209°. ¹³³⁸
 S[CH₂CH₂N(C₁₆H₃₃)NMe₂]₂Cl₂, m. 219°. ¹⁴⁹⁰

Nitrosulfides

MeSCH₂CH₂NO₂, b₂₀ 105°. ⁶¹⁸
 MeSCH₂CHMeNO₂, b₁₆ 98–100°. ⁶¹⁸
 BuSCH₂CHMeNO₂, b₁₃ 124–8°. ⁶¹⁸

$\text{PhSCHMeCH}_2\text{NO}_2$, $b_{0.1}$ 110–1°.^{618, 619}
 $\text{BuSCH}_2\text{CHEtNO}_2$, b_1 82–6°; d 25/4 1.0143; n 25/D 1.470.⁶⁹
 $t\text{-BuSCH}_2\text{CHEtNO}_2$, b_3 89–93°; d 25/4 1.007; n 25/D 1.469.⁶⁹
 $\text{PhCH}_2\text{SCHEtCH}_2\text{NO}_2$, b_2 129–31°; n 25/D 1.5453.¹⁰⁷⁵
 MeSCHMeCHMeNO_2 , b_{15} 91–6°.^{618, 619}
 BuSCHEtCHEtNO_2 , b_2 93°; d 25/4 0.9881; n 25/D 1.470.⁶⁹
 $t\text{-BuSCHPrCHEtNO}_2$, $b_{0.3}$ 75–6°; d 25/4 0.9976; n 25/D 1.473.⁶⁹
 $\text{BuSCMe}_2\text{CH}_2\text{NO}_2$, b_{11} 124°.⁶¹⁸
 $\text{PhCH}_2\text{SCMe}_2\text{CH}_2\text{NO}_2$, b_3 137–8°; n 25/D 1.5540.³²⁸
 $\text{MeSCHPhCH}_2\text{NO}_2$, b_{22} 168–72°.⁶¹⁸
 $\text{PrSCHPhCH}_2\text{NO}_2$, b_{11} 111–2°.¹³⁸⁰
 $t\text{-BUSCHPhCH}_2\text{NO}_2$, m . 65°.¹³⁸⁰
 $\text{PhSCHPhCH}_2\text{NO}_2$, m . 73°.²⁶³
 $\text{MeC}_6\text{H}_4\text{SCHPhCH}_2\text{NO}_2$, *ortho*, m . 62.5°; *para*, m . 77°.²⁶³
 $i\text{-PrC}_6\text{H}_4\text{SCHPhCH}_2\text{NO}_2$, *p*, m . 75°.²⁶³
 $\text{PhCH}_2\text{SCHPhCH}_2\text{NO}_2$, m . 39°.⁶⁹
 $\beta\text{-C}_{10}\text{H}_7\text{SCHPhCH}_2\text{NO}_2$, m . 115°.⁶⁹
 PhSCHPhCHMeNO_2 , m . 65.6°.²⁶³
 $\text{PhCH}_2\text{SCH}(\text{CH}_2\text{Ph})\text{CH}_2\text{NO}_2$, $b_{1.5}$ 98–9°; n 18.5/D 1.5080.¹⁰⁷⁵
 $\text{PhSCH}(\text{C}_6\text{H}_4\text{CHMe}_2\text{-}p)\text{CH}_2\text{NO}_2$, m . 77°.²⁶³
 $\text{PhSCH}(\text{C}_6\text{H}_4\text{OMe}_2\text{-}p)\text{CH}_2\text{NO}_2$, m . 101°.²⁶³
 $p\text{-MeC}_6\text{H}_4\text{SCH}(\text{C}_6\text{H}_4\text{OMe-}p)\text{CH}_2\text{NO}_2$, m . 74°.²⁶³
 $\text{PhSCH}(\text{C}_6\text{H}_3\text{O}_2\text{CH}_2\text{-}3,4)\text{CH}_2\text{NO}_2$, m . 71°.²⁶³
 $\text{MeSCH}_2\text{CHMe}(\text{NO}_2)\text{CH}_2\text{CH}(\text{NO}_2)\text{Me}$, $b_{0.05}$ 110–5°.^{823, 824}
 $\text{S}(\text{CH}_2\text{CH}_2\text{NO}_2)_2$, $b_{0.2}$ 140–4°.⁶¹⁸
 $\text{S}(\text{CH}_2\text{CHEtNO}_2)_2$, b_1 139°; d 25/4 1.170; n 25/D 1.489.⁶⁹
 $\text{S}(\text{CHEtCH}_2\text{NO}_2)_2$, b_1 155°; d 25/4 1.171; n 25/D 1.492.⁶⁹
 $\text{S}(\text{CHPhCH}_2\text{NO}_2)_2$, m . 108°.⁶⁹
 $\text{S}(\text{CMe}_2\text{CH}_2\text{NO}_2)_2$, m . 60°.⁶¹⁸

Nitro-Halo-Sulfides

NITRO-CHLOROSULFIDES

$p\text{-ClC}_6\text{H}_4\text{SCHPhCH}_2\text{NO}_2$, m . 64°.²⁶³
 $p\text{-ClCH}_2\text{CH}_2\text{SCH}_2\text{C}_6\text{H}_4\text{NO}_2$, m . 50°.^{106b}
 $\text{MeSC}_6\text{H}_3\text{ClNO}_2\text{-}4,2$, m . 130°.¹²⁹⁰ 129°.^{656a} 128°.^{140a}
 $\text{MeSC}_6\text{H}_3\text{ClNO}_2\text{-}5,2$, m . 130°.^{656a}
 $\text{MeSC}_6\text{H}_3\text{ClNO}_2\text{-}3,4$, m . 61°.^{656b}
 $\text{EtSC}_6\text{H}_3\text{ClNO}_2\text{-}4,2$, m . 99.5°.¹³⁰⁷
 $\text{ClCMe}_2\text{CH}_2\text{SC}_6\text{H}_3\text{ClNO}_2\text{-}4,2$, m . 80.2°.¹⁴⁰¹

- $2\text{-ClC}_5\text{H}_8\text{SC}_6\text{H}_3\text{ClNO}_2\text{-4,2}$, m. 60° .¹⁴⁰¹
 $2\text{-ClC}_6\text{H}_{10}\text{SC}_6\text{H}_3\text{ClNO}_2\text{-4,2}$, m. 79.6° .¹⁴⁰¹
 $\text{Cl}_3\text{CSC}_6\text{H}_3\text{ClNO}_2\text{-4,2}$, m. 61° .^{1093.5}
 $\text{PhCH}_2\text{SC}_6\text{H}_3\text{ClNO}_2\text{-2,4}$, m. 109° .¹¹⁷⁸
 $\text{PhCH}_2\text{SC}_6\text{H}_3\text{ClNO}_2\text{-4,2}$, m. 132.5° .¹³⁰⁷
 $\text{PhCH}_2\text{SC}_6\text{H}_3\text{ClNO}_2\text{-2,6}$, m. 62° .¹³⁰⁷
 $\text{MeC}_6\text{H}_4\text{SC}_6\text{H}_3\text{ClNO}_2\text{-2,4}$, *ortho*, m. 82.5° ; *para*, m. 121° .¹⁴⁰⁸
 $p\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_3\text{ClNO}_2\text{-2,6}$, m. 70° .⁸⁷²
 $\text{PhCH}_2\text{SC}_6\text{H}_2\text{Cl}_2\text{NO}_2\text{-4,5,2}$, m. 101.5° .¹³⁰⁷
 $4,2\text{-H}_2\text{NCIC}_6\text{H}_3\text{SC}_6\text{H}_3\text{ClNO}_2\text{-2,4}$, m. 211° ; Ac., m. 187° .⁶⁵⁵
 $\text{S}(\text{C}_6\text{H}_3\text{ClNO}_2\text{-2,4})_2$, m. 166° .⁶⁵⁵ 163° .¹²⁵
 $\text{S}(\text{C}_6\text{H}_3\text{ClNO}_2\text{-4,2})_2$, m. 149.5° .⁹⁹ 149° .¹¹⁸⁰ 146° .⁶⁵⁸
 $\text{S}(\text{CH}_2\text{C}_6\text{H}_3\text{ClNO}_2\text{-2,5})_2$, m. 119° .¹³⁰⁸
 $\text{S}(\text{C}_6\text{H}_2\text{Cl}_2\text{NO}_2\text{-2,4,6})_2$, m. $70\text{--}80^\circ$.^{140b}

NITRO-BROMOSULFIDES

- $\text{MeSC}_6\text{H}_3\text{BrNO}_2\text{-4,2}$, m. 131° .²⁹¹ 126° .^{140a}
 $\text{EtSC}_6\text{H}_3\text{BrNO}_2\text{-4,2}$, m. 118.5° .¹³⁰⁷
 $\text{PhCH}_2\text{SC}_6\text{H}_3\text{BrNO}_2\text{-4,2}$, m. 131.5° .¹³⁰⁷
 $p\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_3\text{BrNO}_2$, m. 124° .¹⁴⁰⁸
 $\text{S}(\text{C}_6\text{H}_3\text{BrNO}_2\text{-4,2})_2$, m. 165° .^{140a}

NITRO-IODOSULFIDES

- $\text{PhSC}_6\text{H}_3\text{I}_2\text{NO}_2\text{-2,6,4}$, m. 105.5° .⁸⁹
 $p\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_2\text{I}_2\text{NO}_2\text{-2,6,4}$, m. 122° .⁸⁹
 $p\text{-MeOC}_6\text{H}_4\text{SC}_6\text{H}_2\text{I}_2\text{NO}_2\text{-2,6,4}$, m. 139° .¹²²⁴
 $p\text{-EtOC}_6\text{H}_4\text{SC}_6\text{H}_3\text{INO}_2\text{-2,4}$, m. 152° .¹⁴⁴
 $p\text{-EtOC}_6\text{H}_4\text{SC}_6\text{H}_2\text{I}_2\text{NO}_2\text{-2,6,4}$, m. 175° .¹⁴⁴
 $2,4\text{-PhCH}_2\text{O}(\text{O}_2\text{N})\text{C}_6\text{H}_3\text{SC}_6\text{H}_3\text{INO}_2\text{-2,4}$, m. 138.2° .^{21b}
 $4,2\text{-O}_2\text{N}(\text{OH})\text{C}_6\text{H}_3\text{SC}_6\text{H}_3\text{INO}_2\text{-2,4}$, m. 228° .^{21b}
 $4,2\text{-O}_2\text{N}(\text{H}_2\text{N})\text{C}_6\text{H}_3\text{SC}_6\text{H}_3\text{INO}_2\text{-2,4}$, m. 213.5° .⁹⁶⁸
 $\text{S}(\text{C}_6\text{H}_3\text{INO}_2\text{-2,4})_2$, m. 186° .¹²⁵
 $\text{S}(\text{C}_6\text{H}_3\text{INO}_2\text{-4,2})_2$, m. 196.8° .⁹⁶⁸

Nitro-Hydroxy-Sulfides

- $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{C}_6\text{H}_4\text{NO}_2\text{-}p$, m. 37.5° .^{106b}
 $\text{MeSC}_6\text{H}_2\text{Me}(\text{NO}_2)\text{OH-3,5,4}$, m. 79° ; Ac., m. 70° .¹⁵⁰³
 $p\text{-HOC}_6\text{H}_4\text{SC}_6\text{H}_3\text{MeNO}_2\text{-4,2}$, m. 128° ; Ac., m. 90° .^{1515a}
 $\text{HOCMe}_2\text{CH}_2\text{SC}_6\text{H}_3\text{ClNO}_2\text{-4,2}$, m. 90.4° .¹⁴⁰¹
 $p\text{-HOC}_6\text{H}_4\text{SC}_6\text{H}_3\text{ClNO}_2\text{-4,2}$, m. 130° .¹⁵⁰²

- p -HOC₆H₄SC₆H₃ClNO₂-2,5, m. 167°. ⁸¹²
 4,2-HOClC₆H₃SC₆H₃ClNO₂-2,5, m. 186°. ⁸¹²
 2,4,5-(HO)₂MeC₆H₂SC₆H₃ClNO₂-2,5, m. 198°. ⁸¹²
 S(C₆H₃(NO₂)OH-4,2)₂, m. 246.4°. ^{21b}
 S(C₆H₂Me(NO₂)OH-5,x,2)₂, m. 194°. ⁵³⁸
 2-(α-HOC₁₀H₇)SC₆H₃MeNO₂-4,2, m. 205°; Ac., m. 149°. ^{1515a}
 1-(β-HOC₁₀H₇)SC₆H₃MeNO₂-4,2, m. 129°; Ac., m. 124°. ^{1515a}
 2-(α-HOC₁₀H₇)SC₆H₃ClNO₂-4,2, m. 155°. ¹⁵⁰²
 x-(β-HOC₁₀H₄)SC₆H₃ClNO₂-4,2, m. 185°. ¹⁵⁰²
 S[α-C₁₀H₅(NO₂)OH-6,2]₂, m. 225°. ⁸
 2,6-(*m*-O₂NC₆H₄S)₂C₁₀H₄(OH)₂-1,3, m. 180°; diAc., m. 110.5°. ⁴⁸³

Nitro-Ether-Sulfides

- MeSC₆H₃(NO₂)OMe-4,2, m. 95°. ⁶⁶⁷
 PhSC₆H₃(NO₂)OMe-4,2, m. 89.2°. ²⁴
 [4,2-MeO(O₂N)C₆H₃]₂S, m. 167°, ¹³⁴⁴ 154°. ^{656c}
 [5,2-MeO(O₂N)C₆H₃]₂S, m. 151°. ^{656c}
 [2,4-MeO(O₂N)C₆H₃]₂S, m. 205°. ⁴⁴⁷
 [3,4-MeO(O₂N)C₆H₃]₂S, m. 81°. ⁶⁶⁷
 [2,4-PhCH₂O(O₂N)C₆H₃]₂S, m. 166°. ^{21b}

Nitro-Aminosulfides

- p -AcNHC₆H₄SCHPhCH₂NO₂, m. 123°. ²⁶³
 MeSC₆H₃(NH₂)NO₂-2,4, m. 105°; Ac., m. 157°. ⁶⁵⁴
 MeSC₆H₃(NH₂)NO₂-3,4, m. 116°. ^{656b}
 PhSC₆H₃(NH₂)NO₂-4,2, m. 138°. ¹⁰⁷⁸
 p -MeC₆H₄SC₆H₃(NH₂)NO₂-4,2, m. 152°. ¹⁰⁷⁸
 p -ClC₆H₄SC₆H₃(NH₂)NO₂-4,2, m. 147°. ¹⁰⁷⁸
 MeSC₆H₃Me(NH₂)NO₂-2,5,4, m. 163°. ¹⁵¹⁶
 p -Me₂NC₆H₄SC₆H₃MeNO₂-4,2, m. 175°. ^{1515a}
 4,3-H₂NMeC₆H₃SC₆H₃MeNO₂-3,4, m. 104°; HCl, m. 187°; Ac., m. 143°; Bz., m. 119°. ^{655.5}
 p -H₂NC₆H₄SC₆H₃ClNO₂-4,2, m. 129°; HCl, m. 225°. ^{996b}
 4,3-H₂NMeC₆H₃SC₆H₃ClNO₂-4,2, m. 115°; HCl, m. 220°. ^{996b}
 [2,4-H₂N(O₂N)C₆H₃]₂S, m. 218°. ⁶⁵⁴
 α-(β-H₂NC₁₀H₆)SC₆H₃MeNO₂-4,2, m. 150°; Ac., m. 136°. ^{1515a}
 α-H₂NC₁₀H₅(SC₆H₄NO₂-*o*)₂-2,4, m. 194°; Ac., m. 215°. ¹⁵⁰⁷
 α-H₂NC₁₀H₅(SC₆H₄NO₂-*p*)₂-2,4, m. 171°. ¹⁵¹³
 α-H₂NC₁₀H₆(SC₆H₃MeNO₂-4,2)₂-2,4, m. 137° and 174°; Ac., m. 210°. ^{1515a}

TABLE 5.7
Nitrophenyl Sulfides, $RSC_6H_4NO_2$

<i>R</i>	<i>Ortho</i> M.p. (°C)	<i>Meta</i> M.p. (°C)	<i>Para</i> M.p. (°C)
Cl ₃ C—	—	—	54° 1510b
HOCH ₂ CH ₂ —	100° 106b	42.5° 106b	62° 106b 59° 1426
HOCH ₂ CH ₂ CH ₂ —	—	—	41.5° 106b
HOCH ₂ CH(OH)CH ₂ —	—	—	93.5° 1339
AcOCHClCH ₂ —	—	—	65° 715
AcOCHClCHCl—	—	—	83° 715
AcOCHBrCHBr—	—	—	52–6° 715
ClCH ₂ CH ₂ —	50° 769 52° 834 51° 106b	31° 106b	62° 106b
ClCH ₂ CH ₂ CH ₂ —	—	—	50° 106b
Cl ₃ CCH(OH)—	—	—	66°; Ac., m. 69° 453
BrCH ₂ CH ₂ —	63° 106b	32° 106b	59° 106b 58° 1426
ICH ₂ CH ₂ —	64° 106b	—	67° 106b
2-ClC ₅ H ₈ —	—	—	50.6° 1401
2-ClC ₆ H ₁₀ —	102° 769	—	65.8° 1401
Me ₂ NCH ₂ CH ₂ —	—	—	HCl 217° 454a
Et ₂ NCH ₂ CH ₂ —	—	—	HCl 174° 454a 171.5° 821
Et ₂ NCH ₂ CH ₂ CH ₂ —	—	—	HCl 148° 454a
O ₂ NCH ₂ —	71° 768a	—	—
O ₂ NCHMe—	58° 768a	—	—
O ₂ NCHEt—	73° 768a	—	—

O ₂ NCMe ₂ —	82° 768a	—	—
MeCOCH ₂ —	81° 1507	—	71° 1513
(MeCO) ₂ CH—	137° 207	—	—
<i>p</i> -HOC ₆ H ₄ —	152° ⁹⁴⁰ 131° 1507	83.5°; Ac., 67° 483	154° ¹⁵¹³ 151° 482
PhCOCH ₂ —	141° 769	—	118° 1426
2,5-HOClC ₆ H ₃ —	—	—	154° 1094
3,5,4-Br ₂ (HO)C ₆ H ₂ —	—	137° 483	156° 482
2,4-Me(HO)C ₆ H ₃ —	—	—	193.5°; Ac., 99° 482
2,3,5,4-MeBr ₂ (HO)C ₆ H—	—	—	205° 482
2,4,5-Me(HO)(Me ₂ CH)C ₆ H ₂ —	—	—	Ac., 78° 482
2,3,4,5-MeBr(HO)(Me ₂ CH)C ₆ H—	—	—	127° 482
2,4-(HO) ₂ C ₆ H ₃ —	151° 1507	151.5° 483	185° ⁴⁸² 184° 228b
2,4-(AcO) ₂ C ₆ H ₃ —	103° 1507	78° 483	182° ¹⁵¹³ 180° 227
3,5,2,4-Br ₂ (HO) ₂ C ₆ H—	—	—	115° ¹⁵¹³ 111° 482
2,4,6-(HO) ₃ C ₆ H ₂ —	—	—	180° 482
<i>p</i> -MeOC ₆ H ₄ —	95° 1078	—	245° 228b
<i>p</i> -EtOC ₆ H ₄ —	—	—	71° 89
<i>p</i> -PhCH ₂ OC ₆ H ₄ —	—	106° 483	96° ⁸²⁹ 95° 144
2,4-(MeO) ₂ C ₆ H ₃ —	—	—	—
3,5-(MeO) ₂ C ₆ H ₃ —	—	—	126° 228a
<i>o</i> -ClC ₆ H ₄ —	122° 1078	—	74°; b ₁ 220–5° 228b
<i>m</i> -ClC ₆ H ₄ —	108° 1078	—	114° 556a
<i>p</i> -ClC ₆ H ₄ —	94° 1078	—	71.5° 556a
<i>m</i> -BrC ₆ H ₄ —	108° 1078	—	88° ^{228b} 87° ^{793a} 84° 556a
<i>p</i> -BrC ₆ H ₄ —	—	—	73° ¹⁴⁶⁶ 72° 1078
			94° 23

TABLE 5.7 (Continued)

<i>R</i>	<i>Ortho</i> M.p. (°C)	<i>Meta</i> M.p. (°C)	<i>Para</i> M.p. (°C)
<i>m</i> -IC ₆ H ₄ —	100° 1078	—	82° 1078
<i>p</i> -IC ₆ H ₄ —	131° 1078	—	102° 22, 23
<i>o</i> -H ₂ NC ₆ H ₄ —	86° 940 81.5° 657	—	93° 657
<i>o</i> -AcNHC ₆ H ₄ —	138° 433a	—	—
<i>o</i> -PhSO ₂ NHC ₆ H ₄ —	172° 433a	—	—
<i>o</i> -(<i>o</i> -NO ₂ C ₆ H ₄ CONH)C ₆ H ₄ —	150° 433a	—	—
<i>m</i> -H ₂ NC ₆ H ₄ —	105° 996b	—	—
<i>p</i> -H ₂ NC ₆ H ₄ —	106° 996b 105.5° 940	—	145° 1349 143° 657, 754, 940 142.5° 1195 141° 996b
<i>p</i> -AcNHC ₆ H ₄ —	195° 907	191° 1513	193° 754
<i>o</i> -MeNHC ₆ H ₄ —	110° 433a	—	—
<i>o</i> -AcMeNC ₆ H ₄ —	124° 433a	—	—
<i>o</i> -(<i>o</i> -O ₂ NC ₆ H ₄ CONMe)C ₆ H ₄ —	146° 433a	—	—
<i>p</i> -Me ₂ NC ₆ H ₄ —	188° 1507	120° 1078 116° 833.5	157° 1513
2,5-H ₂ NMeC ₆ H ₃ —	108° 996b	—	—
4,3-H ₂ NMeC ₆ H ₃ —	103° 996b	—	—
2,5-Me ₂ NMeC ₆ H ₃ —	104° 433a	—	—
4,3-H ₂ NCIC ₆ H ₃ —	—	—	129° 996b
4,2-O ₂ NCIC ₆ H ₃ —	—	—	144° 125
4,2-O ₂ NBrC ₆ H ₃ —	—	—	159° 125
2,4-O ₂ NBrC ₆ H ₃ —	131° 140a	—	—

4,2-O ₂ NIC ₆ H ₃ —	—	—	168° 123
4,2-O ₂ N(AcO)C ₆ H ₃ —	—	—	100° 125
α-(2-HOC ₁₀ H ₆)—	180° 1507	106° 483	176° 1513
α-(2-AcOC ₁₀ H ₆)—	101° 1507	85.5° 483	111° 1513
α-(4-HOC ₁₀ H ₆)—	186° 1507	—	172° 1513
α-(4-AcOC ₁₀ H ₆)—	126° 1507	—	85° 1513
α-(2-PhCH ₂ OC ₁₀ H ₆)—	—	137° 483	—
α-(2-H ₂ NC ₁₀ H ₆)—	184° 1507	—	—
α-(4-H ₂ NC ₁₀ H ₆)—	—	—	HCl 168° 1513

TABLE 6.7

*Dinitrophenyl Sulfides, RSC₆H₃(NO₂)₂-2,4 **

R	M.p. (°C)
ClCH ₂ CH ₂ -	94.5° ⁷⁶⁶ 97° ¹¹⁴
ClCH ₂ CHMe-	109.5° ⁷⁶⁶
ClCHMeCH ₂ -	76° ⁷⁶⁶ 77° ^{344b}
ClCMe ₂ CH ₂ -	87° ^{344b}
ClCHMeCHMe-	<i>cis</i> m. 129° ^{768.5}
ClCH ₂ CHClCH ₂ -	111° ⁷⁶⁷
ClCH:CH-	130.5° ⁷⁶⁵
ClCMe:CMc-	76° ⁷⁶⁵
ClCet:CEt-	66° ⁷⁶⁵
BrCH ₂ CH ₂ -	92° ¹¹⁴
BrCHMeCHMe-	<i>cis</i> m. 137°; <i>trans</i> m. 92° ^{768.5}
BrCH ₂ CHClCH ₂ -	111° ⁷⁶⁷
HOCH ₂ CH ₂ -	100° ^{114, 331b} 102° ^{587.5}
HOCH ₂ CHMe-	64° ^{344a} 87° ²⁷⁶
HOCHMeCH ₂ -	94° ^{344a}
BzOCHMeCH ₂ -	139° ²⁷⁶
HOCH ₂ CH(OH)CH ₂ -	143° ^{344b}
ClCH ₂ CH(OH)CH ₂ -	82° ^{344b}
MeOCH ₂ CH ₂ -	90° ²⁷⁶
EtOCH ₂ CH ₂ -	66° ^{171, 872}
(EtO) ₂ CHCH ₂ -	56° ⁶⁴⁵
MeCOCH ₂ -	139° ^{344a} 140.5° ⁷⁶⁹
OC(CH ₂) ₂ -	195° ¹⁷¹ 200° ^{1250a}
$\begin{array}{ c} \text{OCH}_2\text{CHCH}_2- \\ \hline \end{array}$	95° ^{344b}
O ₂ NCH ₂ -	129.5° ^{768a}
O ₂ NCH ₂ CH ₂ -	197° ⁶¹⁸
O ₂ NCHEt-	82.5° ^{768a}
O ₂ NCMe ₂ -	101.5° ^{768a}
O ₂ NCHPr-	123° ¹³⁰⁶
H ₂ NCH ₂ CH ₂ -	94.5° ⁸⁴
Et ₂ NCH ₂ CH ₂ -	188° ⁵⁶²
Et ₂ NCH ₂ CH ₂ CH ₂ -	145° ⁵⁶²
NCCH ₂ -	140.5° ³⁴³
NCCHMe-	109° ³⁴³

* A number of these in which R is unsubstituted have been given in Chapter 2, Volume I under identification of mercaptans.

TABLE 6.7 (Continued)

R	M.p. (°C)
NCCHMeCHMe-	<i>cis</i> 72.5°; <i>trans</i> 76.5° ^{768.5}
2-ClC ₆ H ₁₀	117° ⁷⁶⁹ 118° ¹⁶⁷
2-BrC ₆ H ₁₀ -	118° ⁷⁶⁷
<i>trans</i> -2-HOC ₅ H ₈	116° ^{166a}
<i>trans</i> -2-HOC ₆ H ₁₀ -	135° ^{166a, 974}
PhCHClCH ₂ -	143.5° ¹⁰⁶²
PhCMeClCHMe-	<i>cis</i> 82°; <i>trans</i> 95° ^{324.5}
PhCCl:CPh	207° ⁷⁶⁵
PhCHBrCH ₂ -	143° ⁷⁶⁷
PhOCH ₂ CH ₂ -	145° ¹⁷¹
PhOCH ₂ CH ₂ CH ₂	105° ¹⁷¹
PhOCH ₂ CH ₂ CH ₂ CH ₂ -	75° ¹⁷¹
PhOCH ₂ CH ₂ OCH ₂ CH ₂ -	94° ¹⁷¹
<i>p</i> -ClC ₆ H ₄ CHClCH ₂	151° ⁷⁶⁷
PhCOCH ₂ -	170° ⁷⁶⁹
<i>o</i> -ClC ₆ H ₄ -	138° ¹⁰⁷⁸
<i>m</i> -ClC ₆ H ₄ -	109° ^{556a}
<i>p</i> -ClC ₆ H ₄ -	122° ^{556a} 123° ¹⁷² 124° ²²¹
2,4-Cl ₂ C ₆ H ₃ -	133.5° ¹³⁰⁶
<i>m</i> -BrC ₆ H ₄ -	110.5° ¹⁰⁷⁸
<i>p</i> -BrC ₆ H ₄ -	141° ²²¹ 142° ¹⁷²
<i>m</i> -IC ₆ H ₄ -	120° ¹⁰⁷⁸
<i>p</i> -IC ₆ H ₄ -	140.5° ¹⁰⁷⁸
<i>m</i> -MeOC ₆ H ₄ -	134° ¹³⁵⁹
2,4-(HO) ₂ C ₆ H ₃ -	184° ²²¹
2,4-O ₂ NCIC ₆ H ₃ -	141° ^{140a}
2,4-O ₂ NBrC ₆ H ₃ -	142° ^{140a}
<i>o</i> -H ₂ NC ₆ H ₄ -	152° ¹⁰⁴⁹
<i>p</i> -Me ₂ NC ₆ H ₄ -	176° ²²¹

TABLE 7.7

Picryl Sulfides, RSC₆H₂(NO₂)₃-2,4,6

M.p.(°C)		M.p.(°C)	
MeCHClCH ₂ -	86° ^{344a}	<i>m</i> -ClC ₆ H ₄ -	105.5° ¹⁰⁷⁸
MeCH(OH)CH ₂ -	68° ^{344a}	<i>p</i> -ClC ₆ H ₄ -	133° ¹⁰⁷⁸
<i>o</i> -ClC ₆ H ₄ -	150° ¹⁰⁷⁸	1,3-(picrylS) ₂ C ₆ H ₃ Cl-4,	202° ¹¹⁰⁷
		1,3-(picrylS) ₂ C ₆ H ₂ Cl ₂ -4,6,	274° ¹¹⁰⁸

Aldehyde-Sulfides

- EtSCH₂CHO, *b*₁₁ 58–63°.^{1074.7}
 ClCH₂CH₂SCH₂CHO, *n* 20/D 1.5050.⁵²¹
 HOCH₂CH₂SCH₂CHO, *m*. 57°; ^{1074.5} 2,4-dinitrophenylhydrazone, *m*. 78°.^{1074.7}
 BuSCH₂CHO, 2,4-dinitrophenylhydrazone, *m*. 70°.^{1075.5}
 PhCH₂SCH₂CHO, *b*₈ 125.5–7°; *d* 22/4 1.1105; *n* 22/D 1.5699.⁵³⁶
 O₂NCH₂CHPhSCH₂CHO, 2,4-dinitrophenylhydrazone, *m*. 139.5°.^{1075.5}
 BuSCHMeCHO, *b*₉ 71.5–2.5°.^{1211d}
 PhCH₂SCMe₂CHO, *b*₇ 90–115°;¹³⁶⁴ *b*₁₄ 144–7°; *n* 21/D 1.5450.^{623, 1374}
 MeSCH₂CH₂CHO, *b*. 166°;²⁶⁵ *b*₇ 51°;⁵⁸⁵ *b*₁₀ 56°;¹⁰⁹⁹ *b*₁₁ 52–4°;¹⁰⁹⁸ *b*₁₂ 60°;^{533, 585} *b*₁₇ 66°; *b*₃₀ 80°; ^{904.5} *d*₂₀ 1.036; *n*_D 1.4850.¹⁰⁹⁸
 EtSCH₂CH₂CHO, *b*. 185°;²⁶⁵ *b*₁₀ 60°;^{1211e} *b*₁₁ 58–63°.^{1074.7}
 (PhSCH₂CH₂CHO)₃, *m*. 163°; isomer, *m*. 110°.⁶⁹⁰
 PhCH₂SCH₂CH₂CHO, *b*_{0.3} 96–8°;^{1416a} *b*₁₂ 158°; ²⁶⁵ *d* 25/4 1.1003; ^{1416a} *n* 19/D 1.5650.²⁶⁵
 MeSCHMeCH₂CHO, *b*_{2.5} 40°.²¹⁰
 EtSCHMeCH₂CHO, *b*₂ 160–80°;¹³⁴⁷ *b*₂₄ 92–3°; *n* 20/D 1.4720;⁵⁹⁸ *n* 27/D 1.529.¹³⁴⁷
 PhCH₂SCHMeCH₂CHO, *b*_{0.7} 124–6°;^{1416a} *b*₂ 120–3°;²¹⁰ *b*₁₀ 156–7°; *n* 20/D 1.5523.²⁶⁵
 PhCH₂SCMe₂CH₂CHO, *b*_{0.1} 109–10°; *b*₁₅ 172°; *n* 15/D 1.5485.²⁶⁵
 MeSC₆H₄CHO, *meta*, *b*_{0.4} 90°; *para*, *m*. 76°.⁷¹
p-EtSC₆H₄CHO, *b*. 244–5°.⁹⁹²
p-MeC₆H₄SC₆H₄CHO-*p*, *m*. 69°.⁸²⁹
p-EtOC₆H₄SC₆H₄CHO-*p*, *m*. 83°.⁸²⁹
p-PhCH₂SC₆H₄CHO, *m*. 70°.⁴²⁰
 MeSC₆H₃(Me)CHO-4,2, *m*. 27°; ⁸¹⁴ *b*_{3.5} 129–34°;⁶⁷⁷ *b*₁₂ 151–2°.⁸¹⁴
 S(CHMeCH₂CHO)₂, *b*₈ 135–6°.⁵⁴³
 2-MeSC₁₀H₈CHO-1, *m*. 65°; *b*₂₀ 220°.²³⁷

Keto-Sulfides

- MeSCH₂COMe, *b*. 152.5–3°; *n* 25/D 1.4713.¹⁸⁷
 EtSCH₂COMe, *b*. 168–72°;¹⁸⁷ 170–2°;⁴⁸ *b*₁₅ 61–2°;^{450c} *b*₂₀ 63.5–7.1°; *n* 20/D 1.4710.⁵²³
 PrSCH₂COMe, *b*₄₈ 101–1.5°; *n* 25/D 1.4681.¹⁸⁷
i-PrSCH₂COMe, *b*₄₈ 92–2.5°; *n* 25/D 1.4627.¹⁸⁷

- $\text{BuSCH}_2\text{COMe}$, m. -49.5° ; b_{50} 116° ,^{999b} $115-9^\circ$.¹⁸⁷
 $i\text{-BuSCH}_2\text{COMe}$, b_{48} $109-9.5^\circ$; n 25/D 1.4621.¹⁸⁷
 $t\text{-BuSCH}_2\text{COMe}$, b_{48} $103.5-4.0^\circ$; n 25/D 1.4632.¹⁸⁷
 $\text{AmSCH}_2\text{COMe}$, b_{48} $133-3.5^\circ$; n 25/D 1.4647.¹⁸⁷
 $\text{HexSCH}_2\text{COMe}$, b_{48} $147.5-8.5^\circ$; n 25/D 1.4646.¹⁸⁷
 $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{COMe}$, b_5 100° .^{999b}
 $\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{COMe}$, $b_{0.75}$ $76-85^\circ$.⁵²²
 $\text{PhSCH}_2\text{COMe}$, m. 36° ,⁴⁸ 35° ; b. $268-9^\circ$,³⁶³ $265-7^\circ$,⁴⁸ b_{15} $143-5^\circ$;
d 4/4 1.2444.³⁶³
 $\text{PhCH}_2\text{SCH}_2\text{COMe}$, b_{17} $155-6^\circ$.¹⁴²⁵
 $p\text{-MeC}_6\text{H}_4\text{SCH}_2\text{COMe}$, b_{7-9} $132-5^\circ$,¹⁰⁸⁵ b_{15} $150-1^\circ$; d 11.5/4
1.0980.³⁶³
 $4,2\text{-Me}(\text{O}_2\text{N})\text{C}_6\text{H}_3\text{SCH}_2\text{COMe}$, m. 117° .^{1515a}
 $4,2\text{-Cl}(\text{O}_2\text{N})\text{C}_6\text{H}_3\text{SCH}_2\text{COMe}$, m. 115° .¹⁵⁰²
 $4,2\text{-Br}(\text{O}_2\text{N})\text{C}_6\text{H}_3\text{SCH}_2\text{COMe}$, m. 123.5° .¹¹⁷¹
 $\text{MeSCH}_2\text{COPh}$, b_{12} $131-7^\circ$,¹¹¹⁷ b_2 $102-4^\circ$,⁸⁶⁹ $b_{0.6}$ 100° ,^{151b} $b_{0.8}$
 $130-2^\circ$; ⁵¹³ n 20/D 1.5836.⁸⁶⁹
 $\text{EtSCH}_2\text{COPh}$, m. 36° ; ^{450c} $b_{0.3}$ $104-6^\circ$,¹¹¹⁷ b_5 $116-8^\circ$,^{450c} b_2 106° ;
n 20/D 1.5700.⁸⁶⁹
 $\text{PrSCH}_2\text{COPh}$, b_2 $120-1^\circ$; n 20/D 1.5603.⁸⁶⁹
 $i\text{-PrSCH}_2\text{COPh}$, b_2 107° ; n 20/D 1.5590.⁸⁶⁹
 $\text{BuSCH}_2\text{COPh}$, b_2 133° ,⁸⁶⁹ b_3 140° ; d 0/4 1.0710, d 25/4 1.0560;
n 20/D 1.5050,^{1455b} 1.5513.⁸⁶⁹
 $i\text{-BuSCH}_2\text{COPh}$, b_2 $125-5^\circ$; n 20/D 1.5486.⁸⁶⁹
 $\text{AmSCH}_2\text{COPh}$, b_2 153° ; n 20/D 1.5457.⁸⁶⁹
 $\text{HexSCH}_2\text{COPh}$, b_2 155° ; n 20/D 1.5391.⁸⁶⁹
 $\text{C}_{12}\text{H}_{25}\text{SCH}_2\text{COPh}$, m. 35° ; $b_{0.32}$ $147-8^\circ$.⁹²⁶
 $\text{C}_6\text{H}_{11}\text{SCH}_2\text{COPh}$, b_2 $153-5^\circ$; n 20/D 1.5705.⁸⁶⁹
 $\text{PhSCH}_2\text{COPh}$, m. 54° .⁸⁶⁹
 $\text{PhCH}_2\text{SCH}_2\text{COPh}$, m. 89° ,¹⁴²⁵ 87° .⁸⁶⁹
 $p\text{-MeC}_6\text{H}_4\text{SCH}_2\text{COPh}$, m. 37° .⁵⁶⁰
 $p\text{-ClC}_6\text{H}_4\text{SCH}_2\text{COPh}$, m. 82° .¹⁴²²
 $4,2\text{-ClNO}_2\text{C}_6\text{H}_3\text{SCH}_2\text{COPh}$, m. 155° .¹⁵⁰²
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{COPh}$, m. 111.5° .¹⁴²⁶
 $\text{Et}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{SCH}_2\text{COPh}$, $b_{0.8}$ $158-60^\circ$.⁵¹³
 $\text{MeSCH}_2\text{COC}_6\text{H}_4\text{Me-}p$, $b_{0.15}$ $92-5^\circ$.¹¹¹⁷
 $\text{EtSCH}_2\text{COC}_6\text{H}_4\text{Me-}p$, $b_{0.15}$ $103-6^\circ$.¹¹¹⁷
 $\text{MeSCH}_2\text{COC}_6\text{H}_3(\text{OH})_{2-3,4}$, m. 110° ; diAc., m. 98° .¹¹¹⁷
 $\text{EtSCH}_2\text{COC}_6\text{H}_3(\text{OH})_{2-3,4}$, m. 94.5° ; diBz., m. 108.5° .¹¹¹⁷
 $\text{MeSCH}_2\text{COC}_6\text{H}_4\text{OMe-}p$, m. 133° .¹¹¹⁷

- $\text{EtSCH}_2\text{COC}_6\text{H}_4\text{OMe-}p$, b_{11} 181° .¹¹¹⁷
 $\text{MeSCH}_2\text{COC}_6\text{H}_3(\text{OMe})_{2-3,4}$, m. 53° .¹¹¹⁷
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{COC}_6\text{H}_4\text{NO}_2\text{-}p$, Ac., m. 187° .⁴⁵⁵
 $p\text{-H}_2\text{NC}_6\text{H}_4\text{SCH}_2\text{COC}_6\text{H}_4\text{NH}_2\text{-}p$, m. 142° ; Ac., m. 182° .⁴⁵⁵
 $\text{MeSCH}_2\text{COC}_{10}\text{H}_6\text{-}\beta$, m. 41° ; $b_{0.2}$ $90\text{--}7^\circ$.¹¹¹⁷
 $\text{BuSCH}_2\text{COC}_4\text{H}_9\text{S-}2$, m. 79° .^{778a}
 $\text{PhSCH}_2\text{COC}_4\text{H}_9\text{S-}2$, b_3 $165\text{--}70^\circ$.^{778a}
 $\text{PhCH}_2\text{SCH}_2\text{COC}_4\text{H}_9\text{S-}2$, m. 79° .^{778a}
 $\alpha\text{-(4-AcNHC}_{10}\text{H}_6)\text{SCH}_2\text{COMe}$, m. 160° .¹⁵¹⁸
 MeSCHMeCOPh , $b_{0.2}$ $86\text{--}8^\circ$.¹¹¹⁷
 EtSCHMeCOPh , $b_{0.2}$ $100\text{--}2^\circ$.¹¹¹⁷
 $\text{EtSCMe}_2\text{COMe}$, b_{15} $65\text{--}6^\circ$.^{450a}
 $\text{PhSCMe}_2\text{COMe}$, b_3 102° .^{450a}
 EtSCHPhCOPh , m. 79° .¹³⁷⁰
 $\text{MeSCH}(\text{CH}_2\text{Ph})\text{COPh}$, m. 56° .¹³⁸⁹
 $\text{MeSCH}_2\text{CH}_2\text{COMe}$, b_{22} $77\text{--}8^\circ$.⁵⁵²
 $\text{EtSCH}_2\text{CH}_2\text{COMe}$, b_{12} $78\text{--}9^\circ$,^{450a} b_3 $45\text{--}50^\circ$.¹³⁸⁰
 $\text{CH}_2\text{CH:CH}_2\text{SCH}_2\text{CH}_2\text{COMe}$, b_{10} 96° .^{442.5}
 $\text{PhSCH}_2\text{CH}_2\text{COMe}$, b_{16} $91\text{--}6^\circ$,¹³⁸⁰ b_{10} $145\text{--}8^\circ$.^{442.5}
 $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{COMe}$, $b_{1.8}$ 131.5° ; n 26/D 1.5490.¹³⁴⁷
 $\text{EtSCH}_2\text{CH}_2\text{COPh}$, m. 47° .^{450a}
 $\text{PhSCH}_2\text{CH}_2\text{COPh}$, m. 75° .⁵⁵²
 $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{COPh}$, m. 39° ; ⁵⁵² b_3 150° .⁴⁴¹
 $p\text{-MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{COPh}$, m. 84° .⁵⁵²
 $\text{EtSCMe}_2\text{CH}_2\text{COMe}$, b_3 $45\text{--}50^\circ$.¹³⁸⁰
 $\text{C}_{12}\text{H}_{25}\text{SCMe}_2\text{CH}_2\text{COMe}$, b_4 $176\text{--}8^\circ$; n 20/D 1.4694.¹³²⁵
 $\text{PhCH}_2\text{SCMe}_2\text{CH}_2\text{COMe}$, b_4 $137\text{--}8.5^\circ$; n 20/D 1.5390.⁵⁶
 $\text{EtSCMe}_2\text{CH}_2\text{COCH:CM}_2$, b_3 $81\text{--}3^\circ$.¹³⁸⁰
 $\text{EtSCHPhCH}_2\text{COMe}$, m. 10° ; b_2 96° .¹³⁸⁰
 $\text{PhSCHPhCH}_2\text{COMe}$, m. 59° .¹²¹⁹
 $p\text{-MeC}_6\text{H}_4\text{SCHPhCH}_2\text{COMe}$, m. 64° .¹³⁸⁰
 $\text{MeSCHPhCH}_2\text{COPh}$, m. 48° .^{778b}
 $\text{EtSCHPhCH}_2\text{COPh}$, m. 67° .^{778b}
 $\text{PrSCHPhCH}_2\text{COPh}$, m. $36\text{--}40^\circ$.¹³⁸⁰
 $t\text{-BuSCHPhCH}_2\text{COPh}$, m. 81° .¹³⁸⁰
 $\text{C}_{12}\text{H}_{25}\text{SCHPhCH}_2\text{COPh}$, m. 54° ,⁴⁸⁷ 52° .^{778b}
 $\text{PhSCHPhCH}_2\text{COPh}$, m. 120° .^{946a}
 $\text{PhCH}_2\text{SCHPhCH}_2\text{COPh}$, m. 71° .^{1038b}
 $p\text{-MeC}_6\text{H}_4\text{SCHPhCH}_2\text{COPh}$, m. 114° ,¹³⁸⁰ 113° ,^{1038a} 111° .⁵⁶⁰
 $\text{MeSCH}(\text{C}_6\text{H}_4\text{OMe-}p)\text{CH}_2\text{COPh}$, m. 56° .^{778b}

- $\text{PrSCH}(\text{C}_6\text{H}_4\text{OMe-}p)\text{CH}_2\text{COPh}$, m. 66° .^{778b}
 $\text{C}_{12}\text{H}_{25}\text{SCH}(\text{C}_6\text{H}_4\text{OMe-}p)\text{CH}_2\text{COPh}$, m. 41.5° .^{778b}
 $\text{PhSCH}(\text{C}_6\text{H}_4\text{OMe-}p)\text{CH}_2\text{COPh}$, m. 86.8° .^{778b}
 $\text{PhCH}_2\text{SCH}(\text{C}_6\text{H}_4\text{OMe-}p)\text{CH}_2\text{COPh}$, m. 58° .^{778b}
 $\text{PhSCH}(\text{C}_6\text{H}_4\text{NO}_2\text{-}m)\text{CH}_2\text{COPh}$, m. 106° .⁶⁴⁴
 $p\text{-MeC}_6\text{H}_4\text{SCH}(\text{C}_6\text{H}_4\text{NO}_2\text{-}m)\text{CH}_2\text{COPh}$, m. 104° .⁶⁴⁴
 $p\text{-BrC}_6\text{H}_4\text{SCH}(\text{C}_6\text{H}_4\text{NO}_2\text{-}m)\text{CH}_2\text{COPh}$, m. 131° .⁶⁴⁴
 $p\text{-MeOC}_6\text{H}_4\text{SCH}(\text{C}_6\text{H}_4\text{NO}_2\text{-}m)\text{CH}_2\text{COPh}$, m. $72\text{--}5^\circ$.⁶⁴⁴
 $t\text{-BuSCHPhCH}_2\text{COCH:CHPh}$, m. 101° .¹³⁸⁰
 $i\text{-AmSCHPhCH}_2\text{COCH:CHPh}$, m. 61° .¹²¹⁹
 $\text{PhSCHPhCH}_2\text{COCH:CHPh}$, m. 128° .¹²¹⁹
 $i\text{-AmSCH}(\text{CH:CHPh})\text{CH}_2\text{COPh}$, m. 64° .¹²¹⁹
 $\text{PhSCH}(\text{CH:CHPh})\text{CH}_2\text{COPh}$, m. 104° .¹²¹⁹
 $p\text{-MeC}_6\text{H}_4\text{SCMe}_2\text{CH}_2\text{CH}_2\text{COMe}$, m. 21° .¹³⁸⁰
 $\text{MeSCH}_2\text{CH}_2\text{CHPhCOPh}$, b_4 $171\text{--}2^\circ$.⁷⁴⁵
 $2\text{-MeS-cyclohexanone}$, b_{14} $109\text{--}12^\circ$.^{202.5}
 $3\text{-PhS-5,5-Me}_2\text{-2-cyclohexenone}$, m. 51° .²⁴⁹
 $p\text{-MeSC}_6\text{H}_4\text{COMe}$, m. 83° ,⁵⁴ 82° ,⁸¹⁴ 79° ,²⁴³ 75° ; ³²² b_{10} 170° ,²⁴³
 b_{12} $154\text{--}6^\circ$.¹⁰⁵⁷
 $p\text{-MeSC}_6\text{H}_4\text{COEt}$, m. 63° ,²⁴³ 61° ; b_{20} 184° ; oxime, m. 101° .⁶⁸⁰
 $p\text{-MeSC}_6\text{H}_4\text{COPr}$, m. 67° ; ²³⁶, ²⁴³ b_{12} 187° .²³⁶
 $p\text{-MeSC}_6\text{H}_4\text{COBu}$, m. 54° ; semicarbazone, m. 177° .²⁴³
 $p\text{-MeSC}_6\text{H}_4\text{COPh}$, m. 79° ,²⁴³, ¹²⁴⁵ 78° ,⁷⁴ 73° ; ¹⁹⁴ b_{11} 217° .²⁴³
 $p\text{-MeSC}_6\text{H}_4\text{COCH}_2\text{Ph}$, m. 101° ,²⁴³ 98° .¹⁴⁸⁸
 $p\text{-EtSC}_6\text{H}_4\text{COMe}$, m. 43.5° ; ^{49.5} b_{14} 167° ; ¹⁰⁵⁷ oxime, m. 91° .^{49.5}
 $p\text{-EtSC}_6\text{H}_4\text{COPh}$, m. 83° ; oximes, m. 134° and 96° ; Ac., m. 100°
and 60° resp.^{49.5}
 $p\text{-PhSC}_6\text{H}_4\text{COMe}$, m. 65° ,³⁸⁰ 68° ; ¹³⁵⁰ b_1 180° .³²²
 $4,2\text{-Me}(\text{O}_2\text{N})\text{C}_6\text{H}_3\text{SC}_6\text{H}_4\text{COMe}$, m. 128° .^{1515a}
 $p\text{-PhSC}_6\text{H}_4\text{COPh}$, m. 72° .³⁸⁰
 $p\text{-PhSC}_6\text{H}_4\text{COCH:CHPh}$, m. 95° .³⁸⁰
 $p\text{-PhSC}_6\text{H}_4\text{COCH:CHC}_6\text{H}_4\text{Me-}p$, m. 110° .³⁸⁰
 $4,2\text{-MeS}(\text{Me})\text{C}_6\text{H}_3\text{COMe}$, b_2 $125\text{--}6^\circ$; n 25/D 1.6090.¹³⁵⁷
 $2,5\text{-MeS}(\text{Me})\text{C}_6\text{H}_3\text{COEt}$, m. 43° ; b_{10} $176\text{--}7^\circ$.⁸¹⁵
 $4,2\text{-MeS}(\text{Me})\text{C}_6\text{H}_3\text{COCH}_2\text{Br}$, m. 86° .¹³⁵⁷
 $2,5\text{-MeS}(\text{Me})\text{C}_6\text{H}_3\text{COCHMeBr}$, m. 98° .⁸¹⁵
 $2,5\text{-MeS}(\text{Me})\text{C}_6\text{H}_3\text{COCHMe}_2$, b_{12} $159\text{--}60^\circ$.⁸¹⁴
 $2,5\text{-MeS}(\text{Me})\text{C}_6\text{H}_3\text{COPh}$, b_1 $172\text{--}4^\circ$.⁸¹⁴
 $2,5\text{-MeC}_6\text{H}_4\text{S}(\text{Me})\text{C}_6\text{H}_3\text{COMe}$, m. 120.5° .⁸¹⁶
 $2,5\text{-MeC}_6\text{H}_4\text{S}(\text{Me})\text{C}_6\text{H}_3\text{COEt}$, m. 66° .⁸¹⁶

2,5-MeC₆H₄S(Me)C₆H₃COCH₂Ph, m. 101.5°. ⁸¹⁶

p-EtSC₆H₄COCHN₂, m. 75°. ⁵¹⁶

p-BuSC₆H₄COCHN₂, m. 79°. ⁵¹⁶

Bis-Sulfide-Ketones

(MeSCH₂)₂CO, b₉ 106–9°. ^{1250c}

(EtSCH₂)₂CO, b₁₅ 117–21°, ¹¹⁹³ b₁₁ 123–4; semicarbazone, m. 120°. ^{1250c}

(PhSCH₂)₂CO, m. 43°; ^{1250c} b₁₂ 256–60°; ⁹¹³ semicarbazone, m. 117°. ^{1250c}

(EtSCMe₂CH₂)₂CO, b₃ 102–5°. ¹³⁸⁰

(HOCH₂CH₂SCMe₂CH₂)₂CO, b₁ 140–5°. ¹³⁸⁰

(HO₂CCH₂SCMe₂CH₂)₂CO, m. 115°. ¹³⁸⁰

(EtSCHPhCH₂)₂CO, m. 35°. ¹³⁸⁰

(PhSCHPhCH₂)₂CO, m. 135°. ¹²¹⁹

(*p*-MeC₆H₄SCHPhCH₂)₂CO, m. 91°. ¹³⁸⁰

(*p*-MeSC₆H₄)₂CO, m. 125.5°, ¹²⁴⁵ 125°. ^{1351.5}

(*p*-EtSC₆H₄)₂CO, m. 118°. ^{1351.5}

(*p*-PhSC₆H₄)₂CO, m. 139°. ³⁷⁹

(*p*-MeOC₆H₄SC₆H₄)₂CO, m. 122°. ³⁷⁹

(*p*-EtOC₆H₄SC₆H₄)₂CO, m. 171°. ³⁷⁹

(MeS)₂C:CHCOPh, m. 94°. ⁷⁵⁸

(MeS)₂C:CHCOC₆H₄Me-*p*, m. 105°. ⁷⁵⁹

(MeS)₂C:CHCOC₄H₉S-2, m. 96.5°. ⁷⁵⁹

(PhCH₂S)₂C:CHCOPh, m. 113°. ⁷⁵⁸

(PhCH₂S)₂C:CHCOC₆H₄Me-*p*, m. 112.5°. ⁷⁵⁹

2,5-(EtSCHPh)₂C₅H₆O, m. 59°. ¹³⁸⁰

2,6-(MeS)₂C₆H₈O, b₁₄ 150°. ^{202.5}

2,6-(EtSCHPh)₂C₆H₈O, m. 89°. ¹³⁸⁰

(2,5-Cl₂C₆H₃S)₂CPhCOPh, m. 138°. ²⁰⁸

2,5-Cl₂C₆H₃S·CPh(COPh)SC₆H₃ClOMe-2,5, m. 178°. ²⁰⁸

(EtS)₃C·CO·C(SEt)₃, b₁₅ 53°. ^{202.3}

(ClCHMeS)₃C·CO·C(SCHMeCl)₃, b₂ 70°. ^{202.3}

Bis-Keto-Sulfides

(MeCOCH₂)₂S, m. 49°, ¹⁵⁵ 45°; ^{999b} b₁₀ 130–5°, ⁹²⁹ b₁₃ 115–25°, ⁶⁷⁸ b₁₄ 126°. ¹⁵⁵

(AmCOCH₂)₂S, m. 65°. ⁷¹⁷

(HepCOCH₂)₂S, m. 78°. ⁷¹⁷

(PhCOCH₂)₂S, m. 77°, ⁵⁰³ 588, 813, 1352 10°; b₈ 129–31°; ^{1250b}

- oxime, m. 151° ; ¹³⁵² phenylhydrazone, m. 126° ; ⁵⁰³ di-phenylhydrazone, 147° . ¹³⁵²
- (*p*-ClC₆H₄COCH₂)₂S, dioxime, m. 150.5° . ²⁸⁸
- (*p*-BrC₆H₄COCH₂)₂S, m. 143.1° ; dioxime, m. 180.5° . ²⁸⁸
- (*p*-MeC₆H₄COCH₂)₂S, m. 89.3° ; dioxime, m. 159° . ²⁸⁸
- (MeCOCHMe)₂S, b₈ $109-11^{\circ}$. ^{1250b}
- [PhCOC(:CHPh)]₂S, m. 140° . ³⁷⁸
- MeCOCH₂SCH₂CH₂COEt, b₉ 130° . ⁵⁵²
- (MeCOCH₂CH₂)₂S, b₁₋₂ $108-14^{\circ}$. ⁸⁹⁰
- (PhCOCH₂CH₂)₂S, m. 104° . ⁵⁵²
- (MeCOCH₂CMe₂)₂S, m. 83° . ³⁵
- p*-MeCOC₆H₄SCH(C₆H₄NO₂-*m*)CH₂COPh, m. 97° . ⁶⁴⁴
- 4,3-MeCO(MeO)C₆H₃SCH(C₆H₄NO₂-*m*)CH₂COPh, m. 121° . ⁶⁴⁴
- (*p*-MeCOC₆H₄)₂S, m. 91° . ^{380, 1350}
- (*p*-PhCOC₆H₄)₂S, m. 169° . ³⁸⁰
- (*p*-PhCH:CHCOC₆H₄)₂S, m. 151° . ³⁸⁰
- (*p*-MeOC₆H₄CH:CHCOC₆H₄)₂S, m. 179° . ³⁸⁰
- [3,4-MeCO(AcO)C₆H₃]₂S, m. 66° . ⁷²⁴
- [3,4-MeCO(MeO)C₆H₃]₂S, m. 78° . ⁷²⁴
- [2,4,5-(HO)₂(MeCO)C₆H₂]₂S, m. 210° ; tetraAc., m. 146° ; dioxime, m. 229° . ⁷²⁴
- [2,4,3,5-(HO)₂Br(MeCO)C₆H]₂S, m. 233° . ⁷²⁴
- [4,2,5-(HO)(BzO)(MeCO)C₆H₂]₂S, m. 229° ; diAc., m. 146° . ⁷²⁴
- [4,2,5-(HO)(MeO)(MeCO)C₆H₂]₂S, m. 224° ; oxime, m. 241° ; diAc., m. 148° ; diBz., m. 173° . ⁷²⁴
- [4,2,5-(HO)(PhCH₂O)(MeCO)C₆H₂]₂S, m. 203° ; diAc., m. 175° . ⁷²⁴
- [2,4,5-(MeO)₂(MeCO)C₆H₂]₂S, m. 197° . ⁷²⁴
- [2,4,5-(EtO)₂(MeCO)C₆H₂]₂S, m. 153° . ⁷²⁴
- [4,2,5-(MeO)(PhCH₂O)(MeCO)C₆H₂]₂S, m. 129° . ⁷²⁴
- [2,4,5-(PhCH₂O)₂(MeCO)C₆H₂]₂S, m. 109° . ⁷²⁴
- [4,2,5-MeO(BzO)(MeCO)C₆H₂]₂S, m. 247° . ⁷²⁴
- α, α' -[3,4-MeCO(HO)C₁₀H₅]₂S, m. 200° . ⁹
- 1,3-(MeCOCH₂S)₂C₆H₂Cl₂-4,6, m. 97° . ¹⁵¹²
- m*-(PhCOCH₂S)₂C₆H₄, m. 95° ; dioxime, m. 116° . ^{467a}
- p*-(PhCOCH₂S)₂C₆H₄, m. 146° ; dioxime, m. 174° . ^{467b}
- p, p'*-(MeCOCH₂SC₆H₄)₂, m. 165° . ¹⁵⁰⁴
- ClCHMeSCH(COMe)₂, b₁ 82° . ^{202.3}
- p*-MeC₆H₄SCH(COMe)₂, m. 53° . ²⁰⁸
- p*-ClC₆H₄SCH(COMe)₂, m. 70° . ²⁰⁸

2,5-Cl₂C₆H₃SCH(COMe)₂, m. 97.5°. ²⁰⁸
 2,4-O₂NCIC₆H₃SCH(COMe)₂, m. 117°. ¹⁵⁰²
 EtSCHPhCH(COMe)₂, m. 76°. ¹²¹⁹
i-AmSCHPhCH(COMe)₂, m. 58°. ¹²¹⁹
 PhSCHPhCH(COMe)₂, m. 120°. ¹²¹⁹
 PhCH₂SCHPhCH(COMe)₂, m. 78°. ¹²¹⁹
i-AmSCHPhCH(COMe)COPh, m. 105°. ¹²¹⁹
 PhSCHPhCH(COMe)COPh, m. 141°. ¹²¹⁹
 2-MeSC₆H₃O₂ (quinone), m. 148°. ¹⁵
 2-EtSC₆H₃O₂ (quinone), m. 95°. ¹⁵
 2-PrSC₆H₃O₂ (quinone), m. 88°. ¹⁵
 2-BuSC₆H₃O₂ (quinone), m. 82°. ¹⁵
 2-*i*-BuSC₆H₃O₂ (quinone), m. 93°. ¹⁵
 2-PhCH₂SC₆H₃O₂ (quinone), m. 119°. ¹⁵
 2,3,5,6-(EtS)₄C₆O₂ (quinone), m. 91°. ⁵⁸⁷
 (-CH₂SCH₂CH₂COMe)₂, b₁₀ 160°. ^{442.5}
 (PhSC₆H₄CO)₂, 4,4'-diphenylthiobenzil, m. 105°. ³⁸¹
tris-(MeCHClS)-pentane-2,4-dione, b₁₂ 104-7°. ^{202.3}

TABLE 8.7

Anthraquinone Sulfides *

Melting Points (°C) of Anthraquinone Dithioethers and Disulfones

	1,5-bis-sulfides	1,5-bis-sulfones	1,8-bis-sulfides	1,8-bis-sulfones
Methyl				
methyl	—	—	222°	310°
ethyl	229°	—	210°	220°
propyl	209°	291°	173.5°	211°
<i>i</i> -propyl	184° ⁶⁶²	235° ⁶⁶²	189° ⁶⁶²	—
butyl	173.5°	264°	134°	169°
<i>i</i> -amyl	175°	266°	114°	172°
benzyl	276° ⁶⁶²	280° ⁶⁶²	262° ⁶⁶²	255° ⁶⁶²
Ethyl				
ethyl	226.5° **	269.5°	167.5° **	228°
propyl	188.5°	243.5°	—	—
<i>i</i> -propyl	163° ⁶⁶²	213° ⁶⁶²	176° ⁶⁶²	—

* The melting points of the α-anthraquinolyl sulfides, C₁₀H₇O₂SR, have been given in Chapter 2, Volume I, under identification of mercaptans; so only data for the 1,5- and 1,8- disulfides, C₁₀H₆O₂(SR)₂, and C₁₀H₆O₂(SR)(SR) are given. These are from reference 1159 unless otherwise noted.

** According to Gattermann ^{532b} 1,8-Me₂ m. 221°, 1,5-Et₂ m. 230° and 1,8-Et₂ m. 169°.

TABLE 8.7 (Continued)

	1,5-bis-sulfides	1,5-bis-sulfones	1,8-bis-sulfides	1,8-bis-sulfones
butyl	156°	194°	95°	128°
<i>i</i> -amyl	152°	198°	—	—
benzyl	208° ⁶⁶²	210° ⁶⁶²	164° ⁶⁶²	242.5° ⁶⁶²
Propyl				
propyl	227°	265°	142°	210°
<i>i</i> -propyl	133° ⁶⁶²	203° ⁶⁶²	135° ⁶⁶²	—
butyl	175°	220°	119.5°	200.5°
<i>i</i> -amyl	—	—	104°	147.5°
benzyl	210° ⁶⁶²	215° ⁶⁶²	181° ⁶⁶²	227° ⁶⁶²
<i>i</i> -Propyl				
<i>i</i> -propyl	148° ⁶⁶²	222° ⁶⁶²	181° ⁶⁶²	—
butyl	114° ⁶⁶²	186° ⁶⁶²	131° ⁶⁶²	—
<i>i</i> -amyl	97° ⁶⁶²	172° ⁶⁶²	109° ⁶⁶²	—
benzyl	239° ⁶⁶²	229° ⁶⁶²	229° ⁶⁶²	—
Butyl				
butyl	159.5°	184.5°	131°	138°
<i>i</i> -butyl	—	—	103.5°	168.5°
<i>i</i> -amyl	134°	203.5°	116.5°	154°
benzyl	235° ⁶⁶²	228° ⁶⁶²	185° ⁶⁶²	210° ⁶⁶²
<i>i</i> -Amyl				
<i>i</i> -amyl	158.5°	202°	133°	176°
benzyl	211° ⁶⁶²	202° ⁶⁶²	189° ⁶⁶²	201° ⁶⁶²
Benzyl				
benzyl	176° ⁶⁶²	—	189° ⁶⁶²	202° ⁶⁶²
Phenyl				
phenyl	250° ^{532b}	—	—	—

 β -Anthraquinonyl Sulfides ^{532b}

Methyl, m. 162°	Bromoethyl, m. 172°
Ethyl, m. 138°	Dibromoethyl, m. 133.5°
Benzyl, m. 138°	Hydroxyethyl, m. 137°; Ac., m. 128°; Bz., m. 128.5°
Vinyl, m. 133°	Ethoxyethyl, m. 110°
Allyl, m. 126°	Diethoxyethyl, m. 106°

Ethylene, m. 302°
Acetylene, m. 323°

For substituted anthraquinonyl α -sulfides, see reference 532b.

1,4-Bis-Sulfides ^{532b}

Dimethyl, m. 127°	Dibenzyl, m. 230°
Diethyl, m. 177°	

 β -Methyl-1,4-Bis-Sulfide

Diethyl, m. 199–205° ¹²¹⁸

TABLE 9.7
Melting Points ($^{\circ}\text{C}$) of Keto-Hydroxy-Sulfides

ArCOCH(OH)SR ^{778e}

PhCOCH(OH)SR					
Et	m. 80°	$\text{C}_{12}\text{H}_{25}$	m. 81°	Ph	m. 98°
Pr	m. 84°	$\text{C}_{16}\text{H}_{33}$	m. 88°	PhCH_3	m. 74°
<i>i</i> -Pr	m. 49°	$\text{C}_{18}\text{H}_{37}$	m. 91°	<i>p</i> - MeC_6H_4	m. 45°
Hep	m. 81°			$\text{C}_6\text{H}_5\text{OCH}_3$	m. 40°
$p\text{-EtOC}_6\text{H}_4\text{COCH(OH)SR}$					
$\text{C}_{12}\text{H}_{25}$	m. 66°	PhCH_3	m. 65°	$2\text{-C}_6\text{H}_5\text{SCH}_3$	m. 38°
$2\text{-C}_6\text{H}_5\text{O}\cdot\text{COCH(OH)SR}$					
Pr	m. 106°	Hex	m. 95.5°	$\text{C}_{12}\text{H}_{25}$	m. 106°
$2\text{-C}_6\text{H}_5\text{S}\cdot\text{COCH(OH)SR}$					
Me	m. 93°	$\text{C}_{12}\text{H}_{25}$	m. 81°	Ph	m. 56°
Et	m. 59°	$\text{C}_{16}\text{H}_{33}$	m. 91°	PhCH_3	m. 79°
Pr	m. 69°	$2\text{-C}_6\text{H}_5\text{SCH}_3$	m. 55°	$\beta\text{-C}_{10}\text{H}_7$	m. 99°
Hep	m. 77°				

Cyano-Sulfides

MeSCH_2CN , b_{20} 74.5° .⁷⁷⁹
 EtSCH_2CN , b_{13} $72\text{--}3^{\circ}$,^{150a} b_{50} $104\text{--}5^{\circ}$.⁷⁷⁹
 $\text{OctSCHMe}_2\text{CN}$, b_1 $122\text{--}3^{\circ}$.²¹⁴
 $\text{HexCHMeSCMe}_2\text{CN}$, $b_{0.4}$ 83° .²¹⁴
 $2\text{-MeC}_6\text{H}_{10}\text{SCMe}_2\text{CN}$, $b_{0.5}$ $95\text{--}7^{\circ}$.²¹⁴
 PhSCMe_2CN , b_1 $122\text{--}4^{\circ}$.²¹⁴
 $\text{PhCH}_2\text{SCMe}_2\text{CN}$, b_1 $123\text{--}4^{\circ}$.²¹⁴
 $\text{MeSCH}_2\text{CH}_2\text{CN}$, b_{22} $100\text{--}6^{\circ}$,¹³⁴⁷ b_{15} 97° ; d 20/4 1.030; n 20/D 1.4840,⁶⁸⁷ n 28/D 1.4813.¹³⁴⁷
 $\text{EtSCH}_2\text{CH}_2\text{CN}$, b_{21} $108\text{--}9^{\circ}$; d 20/4 1.004; n 20/D 1.4790.⁶⁸⁷
 $i\text{-PrSCH}_2\text{CH}_2\text{CN}$, b_{12} $102.5\text{--}3^{\circ}$; d 20/4 0.968; n 20/D 1.4751.⁶⁸⁷
 $\text{BuSCH}_2\text{CH}_2\text{CN}$, b_{14} $126\text{--}7^{\circ}$; d 20/4 0.956; n 20/D 1.4735.⁶⁸⁷
 $i\text{-BuSCH}_2\text{CH}_2\text{CN}$, b_{30} $132.5\text{--}3^{\circ}$, b_6 106° ; d 20/4 0.948; n 20/D 1.4730.⁶⁸⁷
 $t\text{-BuSCH}_2\text{CH}_2\text{CN}$, b_{17} $113.5\text{--}4^{\circ}$; d 20/4 0.944; n 20/D 1.4733.⁶⁸⁷
 $\text{DecSCH}_2\text{CH}_2\text{CN}$, b_3 $154\text{--}6^{\circ}$; n 20/D 1.4714.¹²⁷⁹
 $\text{C}_{12}\text{H}_{25}\text{SCH}_2\text{CH}_2\text{CN}$, $m.$ 21° ; b_4 $197\text{--}8^{\circ}$,¹²⁷⁹ b_6 $160\text{--}85^{\circ}$; ⁶⁰⁹ n 20/D 1.4709.¹²⁷⁹
 $\text{C}_{18}\text{H}_{37}\text{SCH}_2\text{CH}_2\text{CN}$, $m.$ 51° .¹²⁷⁹
 $\text{ClCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CN}$, b_{15} 155° .¹³³¹
 $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CN}$, b_8 $163.5\text{--}4.5^{\circ}$; d 20/4 1.138; n 20/D 1.5120.⁶⁸⁷

- $\text{PhSCH}_2\text{CH}_2\text{CN}$, b_8 154° , b_{27} $174\text{--}4.5^\circ$; d 20/4 1.114; n 20/D 1.5735.⁶⁸⁷
 $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{CN}$, $b_{0.9}$ 131° ,¹³⁴⁷ b_2 $152\text{--}3^\circ$, b_{19} 192° ; d 20/4 1.088; n 20/D 1.5625,⁶⁸⁷ 1.5655.¹³⁴⁷
 $o\text{-MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{CN}$, b_{16} 177.5° ; d 20/4 1.099; n 20/D 1.5725.⁶⁸⁷
 $m\text{-MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{CN}$, b_{18} 177.5° ; d 20/4 1.083; n 20/D 1.5673.⁶⁸⁷
 $p\text{-MeC}_6\text{H}_4\text{SCH}_2\text{CH}_2\text{CN}$, b_1 $119\text{--}30^\circ$,⁸⁸⁶ b_{17} 178.5° ; d 20/4 1.077; n 20/D 1.5636.⁶⁸⁷
 $2\text{-C}_4\text{H}_9\text{SCH}_2\text{CH}_2\text{CN}$, $b_{2.5}$ $127\text{--}30^\circ$.^{778c}
 $\beta\text{-C}_{10}\text{H}_7\text{SCH}_2\text{CH}_2\text{CN}$, m . 28° ; b_6 $190\text{--}3^\circ$.⁸⁸⁶
 $t\text{-BuSCH}_2\text{CHMeCN}$, $b_{0.04}$ 43° ; n 20/D 1.4662.¹²⁰³
 $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CHMeCN}$, $b_{0.8}$ 102° ; n 20/D 1.5013.¹²⁰³
 $\text{PhSCH}_2\text{CHMeCN}$, $b_{0.04}$ 79° ; n 20/D 1.5598.¹²⁰³
 $\text{PhCH}_2\text{SCH}_2\text{CHMeCN}$, $b_{0.2}$ 89.5° ; n 20/D 1.5515.¹²⁰³
 $\text{PhSCHMeCH}_2\text{CN}$, $b_{0.9}$ 114° ; n 20/D 1.5581.¹²⁰²
 $t\text{-BuSCH}_2\text{CHEtCN}$, $b_{0.11}$ 46° ; n 20/D 1.4672.¹²⁰³
 $\text{PhSCH}_2\text{CHEtCN}$, $b_{0.04}$ 90° ; n 20/D 1.5541.¹²⁰³
 $\text{PhCH}_2\text{SCH}_2\text{CHEtCN}$, $b_{0.2}$ 90° ; n 20/D 1.5456.¹²⁰³
 $t\text{-BuSCH}_2\text{CH}(\text{CHMe}_2)\text{CN}$, $b_{0.2}$ 47° ; n 20/D 1.4713.¹²⁰³
 $\text{PhSCH}_2\text{CH}(\text{CHMe}_2)\text{CN}$, $b_{0.04}$ 90° ; n 20/D 1.5494.¹²⁰³
 $\text{PhCH}_2\text{SCH}_2\text{CH}(\text{CHMe}_2)\text{CN}$, $b_{0.5}$ 97° ; n 20/D 1.5420.¹²⁰³
 $\text{PhSCH}(\text{CHMe}_2)\text{CH}_2\text{CN}$, $b_{0.38}$ 110° ; n 20/D 1.5535.¹²⁰²
 $\text{PhSCH}(\text{CMe}_3)\text{CH}_2\text{CN}$, $b_{0.5}$ 108° ; n 20/D 1.5546.¹²⁰²
 $\text{PhSCH}_2\text{CHAmCN}$, $b_{0.2}$ 115° ; n 20/D 1.5348.¹²⁰³
 $\text{PhCH}_2\text{SCH}_2\text{CHAmCN}$, $b_{0.3}$ 118° ; n 20/D 1.5318.¹²⁰³
 $\text{PhCH}_2\text{SCMe}_2\text{CH}(\text{OH})\text{CN}$, m . 64° .^{623, 1374}
 $\text{PhCH}_2\text{SCMe}_2\text{CH}(\text{NH}_2)\text{CN}$, HCl , m . 152° .¹³⁷⁴
 $\text{MeSCH}_2\text{CH}_2\text{CH}_2\text{CN}$, b . 218° .¹²⁴²
 $\text{PhSCH}_2\text{CH}_2\text{CH}_2\text{CN}$, b_{19} 185° ,²⁴⁴ $b_{0.1}$ $135\text{--}7^\circ$; ⁹⁷ n 26/D 1.5646.²⁴⁴
 $1,1\text{-PhSC}_6\text{H}_{10}\text{CN}$, m . 64.1° .¹²⁰⁴
 $1,1\text{-PhCH}_2\text{SC}_6\text{H}_{10}\text{CN}$, m . 44.0° .¹²⁰⁴
 $\text{MeSC}_6\text{H}_4\text{CN}$, *meta*, m . 40° ; ¹⁵¹⁴ *para*, m . 64° .^{1510b}
 $\text{EtSC}_6\text{H}_4\text{CN}$, *ortho*, b_{15} 170° ; d_{18} 1.110; n 18/D 1.5857; ¹⁹³ *para*, b_{10} $120\text{--}5^\circ$.^{820.5}
 $p\text{-PrSC}_6\text{H}_4\text{CN}$, b_{50} 198° .²⁴⁰
 $p\text{-BuSC}_6\text{H}_4\text{CN}$, b_{24} 200° .²⁴⁰
 $p\text{-MeC}_6\text{H}_4\text{SC}_6\text{H}_4\text{CN}$ -*p*, m . 103° .⁸²⁹
 $p\text{-EtOC}_6\text{H}_4\text{SC}_6\text{H}_4\text{CN}$ -*p*, m . 96° .⁸²⁹
 $\text{MeSC}_6\text{H}_3(\text{Cl})\text{CN}$ -4,2, m . 105° .¹⁸⁹

$\text{Cl}_3\text{CSC}_6\text{H}_3(\text{Cl})\text{CN-4,2}$, m. 85° .^{1093.5}
 $(p\text{-MeC}_6\text{H}_4\text{S})_2\text{CPhCN}$, m. 89° .²⁰⁸
 $(2,5\text{-Cl}_2\text{C}_6\text{H}_3\text{S})_2\text{CPhCN}$, m. 129° .²⁰⁸
 $\text{HOCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{CN}$, $b_{6.5}$ 198° ; d 20/4 1.131; n 20/D 1.4993.⁶⁸⁷
 $\text{PhCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{CN}$, b_{1-2} $177\text{--}85^\circ$; n 25/D 1.53725.³⁵⁴

Bis-Cyano-Sulfides

$\text{S}(\text{CH}_2\text{CN})_2$, m. 47.5° ,¹³²³ 46.5° ,¹⁵²² 46° ; ³⁴³ b_{22} $177\text{--}8^\circ$.¹³²³
 $\text{S}(\text{CH}_2\text{CH}_2\text{CN})_2$, m. 28° ,⁸⁹¹ 1462 25° ; ¹⁰³² b_3 $179\text{--}80^\circ$,^{694b} 763 b_7 $191\text{--}4^\circ$.⁵⁴³
 $\text{S}(\text{CH}_2\text{CMe}_2\text{CN})_2$, m. 66° ; b_5 $155\text{--}6^\circ$.²⁶⁴
 $\text{S}[\text{CH}_2\text{COC}(\text{CN})\text{:C}(\text{OH})\text{Me}]_2$, m. 130° .¹⁰³
 $\text{S}[\text{CH}_2\text{COC}(\text{CN})\text{:C}(\text{NH}_2)\text{Me}]_2$, m. 179° .¹⁰³
 $\text{S}[\text{CH}_2\text{COC}(\text{CN})\text{:C}(\text{NHPh})\text{Me}]_2$, m. 172° .¹⁰³
 $\text{S}[\text{CH}_2\text{COC}(\text{CN})\text{:C}(\text{NH}_2)\text{Ph}]_2$, m. 198° .¹⁰³
 $\text{S}(\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{CN})_2$, b_8 $245\text{--}6^\circ$; d 20/4 1.111; n 20/D 1.4900.⁶⁸⁷
 $\text{NCCH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{CN}$, b_9 225° ; d 20/4 1.199; n 20/D 1.4946.⁶⁸⁷
 $\text{O}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{CN})_2$, d 20/4 1.1283.^{1482a}
 $(\text{CH}_2\text{SCH}_2\text{CH}_2\text{CN})_2$, m. 91.5° .³⁴¹
 $\text{S}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CHMeCN})_2$, $b_{0.1}$ 225° ; n 20/D 1.5485.¹²⁰³

TABLE 10.7

Saccharin Sulfides



R	M.p. (C°)	R	M.P. (C°)	R	M.p. (C°)
Me	219°	Hex	47°	C ₁₁ H ₂₃	80°
Et	184°	Oct	46.5°	C ₁₂ H ₂₅	68°
Pr	103°	Non	73.5°	Ph	219.5°
Bu	92.5°	Dec	60°	PhCH ₂	138°
Am	69.5°				

Silicon Sulfides

- EtSCH₂SiMe₃, *b*₉ 43.5°; *d* 25/4 0.8402; *n* 25/D 1.4512.¹⁰⁴³
 PrSCH₂SiMe₃, *b*₉ 60°; *d* 25/4 0.8377; *n* 25/D 1.4518.¹⁰⁴³
i-PrSCH₂SiMe₃, *b*₉ 53°; *d* 25/4 0.8321; *n* 25/D 1.4494.¹⁰⁴³
 BuSCH₂SiMe₃, *b*₉ 75–5.5°; *d* 25/4 0.8352; *n* 25/D 1.4530.¹⁰⁴³
i-BuSCH₂SiMe₃, *b*₉ 65.5–6°; *d* 25/4 0.8314; *n* 25/D 1.4502.¹⁰⁴³
t-BuSCH₂SiMe₃, *b*₉ 55.5–6.5°; *d* 25/4 0.8720; *n* 25/D 1.4496.¹⁰⁴³
 AmSCH₂SiMe₃, *b*₉ 89°; *d* 25/4 0.8374; *n* 25/D 1.4542.¹⁰⁴³
i-AmSCH₂SiMe₃, *b*₉ 85–6°; *d* 25/4 0.8347; *n* 25/D 1.4530.¹⁰⁴³
 PhCH₂SCH₂SiMe₃, *b*₈ 124°; *d* 25/4 0.9507; *n* 25/D 1.5242.¹⁰⁴³
p-MeC₆H₄SCH₂SiMe₃, *b*₁ 83–5°; *d* 20/4 0.9528; *n* 20/D 1.5356.⁵⁵⁷
p-MeC₆H₄SCH₂SiPh₃, *m.* 149°.⁵⁵⁷
 PhSCH₂CH₂SiPh₃, *m.* 100°.⁵⁵⁷
p-MeC₆H₄SCH₂CH₂SiPh₃, *m.* 99°.⁵⁵⁷
 PhCH₂SCH₂CH₂SiPh₃, *m.* 73°.⁵⁵⁷
p-MeC₆H₄SCH₂CH₂CH₂SiMe₃, *b*₄₈ 206–9°; *d* 20/4 0.9408; *n* 20/D 1.5267.^{206, 222, 223}
 (PhSCH₂)₄Si *m.* 69°.⁵⁵⁷
 (*p*-MeC₆H₄SCH₂)₄Si, *m.* 70°.⁵⁵⁷

Miscellaneous Substituted Sulfides

- p*-MeSC₆H₄SO₂Cl, *m.* 45°.²²⁹
p-MeSC₆H₄SO₂NH₂, *m.* 165°.²²⁹

NITRO-SULFONAMIDES

- MeSC₆H₃(NO₂)SO₂NH₂-2,4, *m.* 223°.¹³⁰⁷
 MeSC₆H₃(NO₂)SO₂NEt₂-2,4, *m.* 147°.¹³⁰⁷

NITRO-SULFINIC ACIDS

- PhSC₆H₃(NO₂)SO₂H-4,2, *m.* 135°.⁸¹²
p-MeC₆H₄SC₆H₃(NO₂)SO₂H-4,2, *m.* 125°.⁸¹²
 2,5-MeOMeC₆H₃SC₆H₃(NO₂)SO₂H-4,2, *m.* 131°.⁸¹²
p-ClC₆H₄SC₆H₃(NO₂)SO₂H-4,2, NH₄ salt, *m.* 115°.⁸¹²
 2,5-Cl₂C₆H₃SC₆H₃(NO₂)SO₂H-4,2, *m.* 28°.⁸¹²

THIOCYANO SULFIDES

- HO(CH₂)₆S(CH₂)₆SCN, *b*_{0.1} 181–6°; *n* 21.5/D 1.507.¹⁴²⁰
 S(CH₂CH₂SCN)₂, *m.* 35°.¹³²³
 NCS(CH₂)₆S(CH₂)₆SCN, *b*_{0.1} 198–210°; *n* 18.5/D 1.5220.¹⁴²⁰

$\text{O}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCN})_2$, d 20/4 1.2465.^{1482a}
 $\text{S}(\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2\text{SCN})_2$, m. 52° .¹⁴⁶²
 $\text{PhCH}_2\text{SCH}_2\text{CH}_2\text{CH}_2\text{NCS}$, $b_{0.2}$ $102-5^\circ$; n 28/D 1.5770.¹³⁴⁷
 $\text{Cl}_3\text{CSC}_6\text{H}_3(\text{Cl})\text{SCN-4,2}$, m. 117° .^{1093.5}

Selenides

$\text{PhCH}_2\text{SeCH}_2\text{Cl}$, m. 38° ; b_8 121° , b_{18} $158-60^\circ$; d 43/4 1.03; n 52/D 1.5587.¹⁴⁶⁵
 $(\text{ClCH}_2)_2\text{Se}$, b. 180° , b_2 42° .²⁰⁰
 $\text{PhSeCH}_2\text{CH}_2\text{OH}$, b_{18} $152-6^\circ$.⁸⁹²
 $\text{PhCH}_2\text{SeCH}_2\text{CH}_2\text{OH}$, b_2 $130-2^\circ$.⁸⁹²
 $(\text{HOCH}_2\text{CH}_2)_2\text{Se}$, decomposes 143° ; d 20/4 1.5830; n 20/D 1.55499.¹²⁹²
 $(\text{MeOCH}_2\text{CH}_2)_2\text{Se}$, b_{772} 218.5° .¹²⁹²
 $\text{PhSeCH}_2\text{CH}(\text{OEt})_2$, b_{17} $153-3.5^\circ$.⁸⁹²
 $(\text{ClCH}_2\text{CH}_2)_2\text{Se}$, m. 24.2° ,^{102, 1226} 25° .⁹²
 $(\text{Cl}_2\text{CHCH}_2)_2\text{Se}$, $b_{0.2}$ $109-14^\circ$.²⁰⁴
 $(\text{Cl}_2\text{CHCHCl})_2\text{Se}$, b. $174-5^\circ$.²⁰⁴
 $\text{Cl}_2\text{CHCH}_2\text{SeCH:CHCl}$, $b_{0.11}$ 115° .²⁰⁴
 $\text{Cl}_2\text{CHCHClSeCCl:CHCl}$, $b_{0.2}$ $104-5^\circ$.²⁰⁴
 $(\text{ClCH:CH})_2\text{Se}$, $b_{0.11}$ $66-9^\circ$.²⁰⁴
 $(\text{ClCH:CCl})_2\text{Se}$, $b_{0.1}$ 75° .²⁰⁴
 $[\text{Cl}_3\text{CCH}(\text{OH})]_2\text{Se}$, m. 98° .⁸⁵³
 $(\text{BrCH}_2\text{CH}_2)_2\text{Se}$, m. 117° .¹⁰²
 $\text{PhSeC}_6\text{H}_4\text{OH}$, *ortho*, $b_{0.5}$ $130-1^\circ$; ⁷⁵⁷ *para*, Ac., m. 55° .⁷⁵⁶
p- $\text{PhSeC}_6\text{H}_4\text{OMe}$, m. 46.3° .⁷⁵⁶
p- $\text{EtSeC}_6\text{H}_4\text{Cl}$, b_3 90° , b_1 85° .⁴⁸⁴
m- $\text{PhSeC}_6\text{H}_4\text{Cl}$, b_{28} $200-5^\circ$.²⁵¹
p- $\text{MeC}_6\text{H}_4\text{SeC}_6\text{H}_4\text{Cl}$ -*p*, m. 73° .²⁵¹
p- $\text{EtSeC}_6\text{H}_4\text{Br}$, b_{18} 146° .⁴⁸⁴
p- $\text{PhSeC}_6\text{H}_4\text{Br}$, m. 33° .⁴¹⁷
 $(2,4\text{-HOMeC}_6\text{H}_3)_2\text{Se}$, m. 111° .¹⁰⁰⁰
 $(4,3\text{-HOMeC}_6\text{H}_3)_2\text{Se}$, m. 99° .¹⁰⁰⁰
 $(p\text{-ClC}_6\text{H}_4)_2\text{Se}$, m. 97° ,⁸⁹² 96° ,⁸⁰⁴ 94° .^{1351b}
 $(p\text{-BrC}_6\text{H}_4)_2\text{Se}$, m. 116° ,²⁷¹ 115.5° ,^{417, 804} 115° .^{1351b}
 $(o\text{-ClC}_6\text{H}_4\text{CH}_2)_2\text{Se}$, m. 54.5° .¹³⁰⁸
 $[p\text{-PhCH}(\text{OH})\text{C}_6\text{H}_4]_2\text{Se}$, m. 114° .³⁸⁰
 $(p\text{-Ph}_2\text{CClC}_6\text{H}_4)_2\text{Se}$, m. 152° .³⁸⁰
 $(p\text{-MeCOC}_6\text{H}_4)_2\text{Se}$, m. 92° .³⁸⁰
 $(p\text{-PhCOC}_6\text{H}_4)_2\text{Se}$, m. 160° .³⁸⁰

- $(p\text{-PhCH:CHCOC}_6\text{H}_4)_2\text{Se}$, m. 156° .³⁸⁰
 $(p\text{-PhCH:CHCH}_2\text{COC}_6\text{H}_4)_2\text{Se}$, m. 166° .³⁸⁰
 $(p\text{-MeOC}_6\text{H}_4\text{CH:CHCOC}_6\text{H}_4)_2\text{Se}$, m. 158° .³⁸⁰
 $[p\text{-3,4}(\text{CH}_2\text{O}_2)\text{C}_6\text{H}_3\text{CH:CHCOC}_6\text{H}_4]_2\text{Se}$, m. 196° .³⁸⁰
 $\text{MeSeC}_6\text{H}_4\text{NHAc}$, *ortho*, m. 105° ; *para*, m. 117° .⁷⁵⁵
 $\text{MeSeC}_6\text{H}_3\text{MeNHAc-4,2}$, m. 117° .⁷⁵⁵
 $\text{MeSeC}_6\text{H}_3\text{MeNHAc-6,2}$, m. 80° .⁷⁵⁵
 $\text{PhSeC}_6\text{H}_4\text{NH}_2$, *ortho*, m. 35° ; ⁷⁵⁷ *para*, m. 94° ; HCl m. 159° ; Ac., m. 170° .⁵³⁷
 $(p\text{-H}_2\text{NC}_6\text{H}_4)_2\text{Se}$, m. 117° ; Ac., m. 219° .¹¹⁴⁵
 $(p\text{-Me}_2\text{NC}_6\text{H}_4)_2\text{Se}$, m. 124° ; picrate, m. 135° .⁵⁷⁰
 $(p\text{-Et}_2\text{NC}_6\text{H}_4)_2\text{Se}$, m. 83° ; HCl, m. 73° ; picrate, m. 135° .⁵⁷⁰
 $p\text{-Me}_2\text{NC}_6\text{H}_4\text{SeC}_6\text{H}_4\text{NO}_2\text{-o}$, m. 169° .⁹⁶
 $p\text{-Me}_2\text{NC}_6\text{H}_4\text{SeC}_6\text{H}_4\text{NO}_2\text{-p}$, m. 180° .⁹⁶
 $(5,2\text{-O}_2\text{NClC}_6\text{H}_3\text{CH}_2)_2\text{Se}$, m. 117.5° .¹³⁰⁸
 $\text{MeSeC}_6\text{H}_4\text{CHO}$, *meta*, m. 10° ; $b_{0.1}$ $78\text{--}9^\circ$; *para*, $b_{0.5}$ 100° .⁷¹
 $(\alpha\text{-MeOC}_{10}\text{H}_8)_2\text{Se}$, m. 138° .^{963.5}
 $(\alpha\text{-EtOC}_{10}\text{H}_8)_2\text{Se}$, m. 149° .^{963.5}
 $o\text{-NO}_2\text{C}_6\text{H}_4\text{SeCH}_2\text{COPh}$, m. 142.5° .¹¹⁷⁰
 $2,4\text{-NO}_2\text{MeC}_6\text{H}_3\text{SeCH}_2\text{COPh}$, m. 113.5° .¹¹⁷⁰
 $2,4\text{-(NO}_2)_2\text{C}_6\text{H}_3\text{SeCH}_2\text{COPh}$, m. 178.5° .¹¹⁷⁰
 $4,2\text{-NO}_2\text{ClC}_6\text{H}_3\text{SeCH}_2\text{COPh}$, m. 156.5° .¹¹⁷⁰
 $4,2\text{-NO}_2\text{BrC}_6\text{H}_3\text{SeCH}_2\text{COPh}$, m. 147.6° .¹¹⁷⁰

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In order to avoid scattering and to bring closely related compounds under one heading, inverted organic names have been used.

For the same reason, it has frequently been necessary to change the name used in the text to a more systematic name.

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